An appraisal of Characteristic Mechanical Properties of Aluminium 6061 alloy – Silicon Carbide (SiCp) Metal Matrix Composite (MMC) exposed to different thermal conditions

A THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE DEGREE OF

Master of Technology

Submitted to

NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

By

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(Roll no: 207MM109)

Under the supervision of

Dr. U.K. Mohanty (Professor)

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ROURKELA-769008, INDIA

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National Institute of Technology, Rourkela.

CERTIFICATE

This is to certify that the Thesis entitled "**An appraisal of Characteristic Mechanical Properties of Aluminium 6061 alloy – Silicon Carbide (SiCp) Metal Matrix Composite (MMC) exposed to different thermal conditions**" submitted by Sri. Sushanta Kumar Sahu(207MM109), Department of Metallurgical and Materials Engineering, NIT, Rourkela, towards a partial fulfillment of requirements for the award of the Degree of Master of Technology, has been carried out under my supervision and has not been submitted elsewhere for award of any degree.

Dr U.K. Mohanty
Professor, Deptt of MME

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ABSTRACT

Aluminium 6061 alloys have been proposed for extensive use in automotive engine applications and there have been discrete cases of experimental implementation. In order to enhance the usability of this material, it has been investigated in composite forms with various ceramic reinforcements. Viability of the different constituents depends on the compatibility of their physical and chemical properties. The service conditions are characterized by extreme stress and temperature conditions very close to failure. Hence thermal stresses play an important role in success of these materials.

The difference in the CTE of the alloy matrix and the ceramic reinforcement results in residual thermal stress build up. It may so cause plastic deformation of the matrix in the vicinity of the reinforcement in order to reduce the residual stresses. However, mismatch in thermal strain values may lead to cracking of the matrix in this process.

In comparison with the matrix and reinforcement, the interface is rather a porous, non-crystalline portion. Therefore residual stresses are released at these sites with relative ease. When the particle fraction is high, interface availability is more; hence, failure of the MMC is due to formation and propagation of cracks at the interfaces. On the contrary, when particle fraction is less, interface availability is poor; failure is predominantly due to particle cracking.

In the present work, cylindrical Al 6061-SiCp MMCs are fabricated in the solid state processing route. The sintering temperature and time of holding at the sintering temperature are varied. The samples are subjected to no thermal shock and thermal shocks at +80 ºC and -80 ºC in different batches. The compressive strengths are determined using an Instron (1195) adopting the ASTM E9 standard for hard metals. Fractured specimen are extensively analyzed with an SEM for failure modes.

Assessment and evaluation on the basis of mechanical properties reveal that at relatively higher sintering temperature and for short term use, the thermal shock is not much damaging. For short- term use, the thermal shock at an elevated temperature is more damaging for samples sintered at lower temperature. For long-term use, the thermal shock due to a sub-ambient temperature is more damaging when test specimen is sintered at relatively a lower temperature.

The micrograph studies reveal that in general when the thermal shock is due to exposure to an elevated temperature, inter-diffusion is high, resulting in strong bonding; hence, the dominating failure mode is cavity generation due to generation of discontinuities at the interface. And when the thermal shock is due to exposure to a sub-ambient temperature, the dominant failure mode is interfacial failure and/or matrix damage.

CHAPTER 1
INTRODUCTION

1. INTRODUCTION

The engineering fraternity has always been on the lookout for wonder-materials which would fit the bills for all types of service conditions. It stem from the need to make progressive discoveries made by scientists, affordable. This affordability quotient has persuaded many researchers to develop such materials which would satisfy various hitherto unexplored conditions. In todays world almost all generic materials have been tried for various uses and their limitations have been met. But the never ending quest of civilization requires that materials qualify for harsher environments. This unavoidable situation demands that new materials be created from various combinations of other compatible materials. It is to be noted here that this method is not new, it has been with mankind since ages. In every part of the world, various materials have been combined to achieve some intended properties, albeit each case differs from the others, i.e. one can create new materials with unique properties, which can be tailor-made and are different from their base ingredients[1]. This concept holds true for a genre of materials called Composite materials where in, various types of matrices may be combined with reinforcements which contribute to the enhancement of the properties. Neither the matrices nor the reinforcements taken alone can stand up to the requirement, but the composite may be able to do so. This alteration in properties can be controlled by many ways, viz. controlling the matrix and reinforcement quality, their proportion or the fabrication route. This flexibility in manufacturing allows one to develop composites with varying properties in a precisely controlled fashion.

It is the superiority of properties that has triggered the penetration of composite materials into all fields of manufacturing. Metal Matrix Composites (MMCs) have emerged as a class of materials suitable for structural, aerospace, automotive, electronic, thermal and wear applications owing to their advantages over the conventional monoliths. They score over in terms of specific modulus, specific strength, high temperature stability, controlled coefficient of thermal expansion, wear resistance, chemical inertness, etc. But the down side is populated by inferior toughness and

high cost of fabrication in comparison with Polymer Matrix Composites (PMCs). But MMCs supersede in terms of higher transverse strength and stiffness, shear strength and high temperature capabilities. The physical properties that attract are no moisture absorption, non-flammability, high electrical and thermal conductivities and resistance to most radiations.

Compositionally, MMCs have at least two components, viz. the matrix and the reinforcement. The matrix is essentially a metal, but seldom a pure one. Except sparing cases, it is generally an alloy. The most common metal alloys in use are based on Aluminium and Titanium. Both of them are low density materials and are commercially available in a wide range of alloy compositions. Other alloys are also used for specific cases, because of their own advantages and disadvantages. Beryllium is the lightest of all structural materials and has a tensile modulus greater than that of steel, but it is extremely brittle, rendering it unsuitable for general purpose use. Magnesium is light, but is highly reactive to Oxygen. Nickel and Cobalt based super alloys have also found some use, but some of the alloying elements present in the matrices have been found to have undesirable effect(promoting oxidation) on the reinforcing fibers at high temperatures.

The reinforcements for MMCs can be broadly divided into five major categories, viz. Continuous fibers, discontinuous fibers, whiskers, wires and particulates. Except the wires being metals, the reinforcements are generally ceramic; which can be oxides, carbides and nitrides which are used because of their excellent combination of specific strengths and stiffness at both ambient and elevated temperatures.

Aluminium alloys have found greater adoption as potential matrix materials in comparison with other alloys. And the 6xxx series of Aluminium alloys are coming into the picture very fast. The aerospace, automotive and utensil industries were the first reporting their use. On the reinforcement side, the most common reinforcement in use is Silicon Carbide (SiC). In the present work, commercial grade Aluminium alloy 6061 (98% assay) and SiCp (99% assay) have been used for the composite preparation.

Metal Matrix Composites, as alternatives to traditional materials, provide the mechanical properties necessary for elevated as well as ambient temperature applications. The performance advantages of these materials include their tailorable mechanical, physical and thermal properties supplemented with light weight, high specific modulus, high strength and thermal conductivity, good fatigue response, control of thermal expansion, and high abrasion and wear resistance. Hence they find use in fabrication of satellite parts, missile, helicopter structures, structural support girders, piston, sleeves and rims, high temperature structures, drive shafts, brake rotors, connecting rods, engine block liners, bike frames, etc.

However, it it is to be clearly understood and acknowledged that the addition of certain volume fraction of a stiff ceramic reinforcement to a ductile matrix (Al 6061 in this case) results in a phenomena specific to reinforced materials. Hence, these issues need to be taken care of: i.e. interfacial bonding between the reinforcement and the matrix, residual stresses, matrix dislocations generated by the thermal mismatch between the phases and reinforcement, and alterations in matrix precipitation kinetics.

The performance limits of MMCs can be enhanced by addition of a high volume fraction of whiskers or particulates. The development of Aluminium MMCs by mixing and consolidating Aluminium alloy powder and the high modulus, low density, micron sized carbides like SiC is one such example. The addition of SiC particulates results in the increase in specific modulus and wear resistance of the MMC, but at the same it it also degrades the ductility and fracture toughness. Thus the addition results in enhancement as well as degradation of the physical and mechanical properties.

S. Pani[24] has investigated the effect of addition of SiCp to commercially pure Aluminium alloys in a pure academic interest. The results have little practical relevance, as the matrix in question is seldom used industrially. Thus, the present work aims to address the shortcomings by using commercially used materials. In the present work, SiCp reinforced Al 6061 (99% assay)

MMCs are fabricated adopting the solid-state processing route, i.e. mixing and consolidation of Al 6061 alloy powder and SiC. The sintering temperature and time at temperature are varied. Some of the test specimen are exposed to +80ºC and -80ºC in different batches. Compressive strength of the treated and untreated samples are calculated with an Instron 1195 adopting the ASTM E9 standard set for Hard Metal Compression test. The load, stress, displacement and strain values at rupture of the samples are compared. These experimental data are tabulated and plotted, and then extensively analyzed for different sintering temperatures and time at sintering temperatures.

The micrographs of the fractured surfaces of the samples as obtained through Scanning Electron Microscope are extensively examined. Attempt has been made to interpret the experimental data by correlating with the inferences of the micrographs obtained. The interfacial bonding, residual stresses generated and matrix dislocations generated as a consequence of thermal mismatch have been focussed to explain the experimental results.

This report contains five numbers of chapters. The first chapter('Introduction') attempts to give an insight to the work undertaken and highlights the procedure adopted in the investigation.

The second chapter ('Literature Survey') is dedicated to an extensive survey of literature related to the present work. Previous works in this field done by other workers, their findings have been revisited and correlated prior to start of the current work. Inferences drawn from these reviews have been used to suitably design and modify the experimental design. Hence, this chapter serves as a base for the next chapter.

The third chapter ('Experimental Design') is devoted to explain the experimental procedure adopted in the present investigation along with the experimental arrangements and details of experimental procedures. The instrument/ apparatus and the prescribed experiments carried out. The instruments/ apparatus and the prescribed experimental norms as adopted in the present investigations have been explained in details.

The fourth chapter('Results and Discussions') contains the results in the form of tables, plots,

bar-diagrams, SEM-micrographs,etc. This also includes a detailed discussion of the results made on the basis of the experimental data.

The ultimate chapter('Conclusions') summarizes the results of the experimental findings.

CHAPTER 2
LITERATURE SURVEY

2.1. Introduction:

Literally the term composite means- a solid material that results when two or more different substances, each with its own characteristics, are combined to create a new substance whose properties are superior to those of the original components for any specific application. The term composite more specifically refers to a structural material within which a reinforcement material (such as silicon carbide) is embedded. And the engineering definition would also go alongside- A material system composed of a mixture or combination of two or more constituents that differ in form or material composition and are essentially insoluble in each other. In principle, composites can be fabricated out of any combination of two or more materials—metallic, organic, or inorganic; but the constituent forms are more restricted. The matrix is the body constituent, serving to enclose the composite and give it a bulk form. Major structural constituents are fibers, particulates, laminates or layers, flakes and fillers. They determine the internal structure of the composite. Usually, they are the additive phase.

When two or more materials are interspersed, there is always a contiguous region. Simply this may be the common boundary of the two phases concerned, in which case it is called an interface. A composite having a single interface is feasibly fabricated when the matrix and the reinforcement are perfectly compatible. On the other end, there may an altogether separate phase present between the matrix phase and the reinforcement phase. This intermediate phase is called an inter-phase. In case there is an inter-phase present, there are two interfaces, one defining the boundary between the matrix and the inter-phase, and the other between the inter-phase and the reinforcement. The strength of the composite in such a case is dependent upon the strength of the weakest of the two interfaces. There are certain advantages of having a preferred inter-phase. Such a composite with an inter-phase is fabricated if the matrix and reinforcement are not chemically compatible or if the wettability of the pair is very poor. such a composite is materialized, by introducing a third material that has good bonding properties, individually with the matrix and the

reinforcement, which would not be possible otherwise.

More or less, the strength of a composite is a function of the strength of its interface between the matrix and the reinforcement. The failure of a functional composite is essentially a result of the failure of the interface. Hence the strengthening mechanism is the most dominant parameter in successful fabrication of a high strength composite[1].

Composites differ by their matrix type, reinforcement type, size and form, composition, temper state, etc. With such a big window available for fabricating a composite from different constituent materials, it is not uncommon to experiment with materials with vividly different properties. There are three broadly classified groups of composites: Polymer Matrix Composite, Metal Matrix Composite and Ceramic Matrix Composite.

2.2. Metal Matrix Composite:

The sustained interest to develop engineering materials which could cope with the raised performance standards, resulted in emergence of a newer class of materials, called Metal Matrix Composites (MMCs). They constitute a family of customizable materials with customizable critical-property relationships. Such materials are known for their exceptional high modulus, stiffness, wear resistance, fatigue life, strength-to-weight ratios, tailorable coefficient of thermal expansion, etc. With these enhancements in properties, they pose for strong candidature for replacing conventional structural materials. But what makes them stand apart is the ability to customize their properties to suit the service requirement. Such advantages have made this group of materials a nice pick for use in weight-sensitive and stiffness-critical components in transportation systems[2].

MMCs can be described as a group of materials in which a continuous metallic phase(matrix) is combined with one or more reinforcement phases. The aim of such a composite material is to enhance the suitability of the end product by selectively enhancing the complimentary properties, and masking the detrimental properties of the matrix and the reinforcement . While

fabricating the MMC, a solid material results when two or more substances are physically (not chemically) combined to create a new material whose properties are superior to those of the original substances for a specific application.

The matrix may be a pure metal or any alloy suitable for the intended application. The reinforcement may be any other material in the form of particulates, whiskers, fibers, platelets, etc. The most common reinforcements are ceramics and metals having nominal size in the range of 0.1 to 100 micrometers. But in fact, just about anything suitable for the application may be utilized as a potential reinforcement. Even though at times, the matrix and the reinforcement both can be metallic in nature, MMCs are not fabricated by conventional alloying methods suitable for metals; since, such a process would mar the essence of a composite. In alloys the phases are not chemically and physically distinct. But in a composite, such phases are intentionally kept distinct, to exploit the properties of the constituents to the fullest.

The reinforcing phase is the nominal constituent of a composite. It is the principal load bearing component in the system. Hence the reinforcements with better mechanical properties than the matrix materials are chosen while designing a composite. The matrix is responsible for holding the load-carrying reinforcement together and retaining the bulk shape of the composite. It also shares some portion of the total load which is transferred to the reinforcement via the interface or vice versa. It is the effectiveness of the interface that decides how much load is transferred to and from the matrix.

In MMCs a high degree of interaction between the matrix and the reinforcement is inherent. The resulting strength is a direct function of effectiveness of the interface between the matrix and the reinforcement. The character of the interface depends upon the chemical and mechanical compatibility of the two phases involved. The chemical incompatibility constraint can be overcome either by opting for a low-temperature processing route or by selecting stable constituents. The thermal-mechanical incompatibility problem is sorted out by employing a ductile matrix that

accommodates the strain generated by the thermal alterations. Also it helps to select a pair of matrix and reinforcement having matching coefficient of thermal expansion. However when it is chemically or thermo-mechanically not feasible to fabricate a composite from a pair of constituents, an intermediate phase which is compatible with the matrix and the reinforcement may be introduced in between the two that masks the incompatibility of the original pair. This inter-phase prevents the chemical reaction between the matrix and the reinforcement and/or aids the matrix in accommodating the strain generated due to any incongruous strain build-up. A soft precipitate-free layer around the reinforcing particulates limit the propagation of the crack generated at their surface by effectively reducing the stress value gradually, thereby increasing the ultimate strength[3].

Metal matrix composites have been under constant development since the days of the World War-II. They were intended to be used in the aircrafts as structural materials. After the war ceased, no longer the purpose was the war, rather MMCs found interest in civilian uses. Today the composites are extensively used in all aspects of life, be it food packaging, medical implants, military armours, automotive applications, space applications or just about anything else. This deep penetration of MMCs in a wide spectrum of application can be attributed to the previously mentioned advantages associated with them.

However, MMCs are not without some drawbacks either. Their inadequate fracture toughness and damage tolerance, poor ductility, size limitations, inhomogeneity of properties, isotropy of properties stand as hindrance to their usability front. Continuous research works are underway to overcome these limitations and explore new possibilities.

Lloyd, D.J.[4], White house et al.[5] and Ribes et al.[6] Studied the effect of particle-induced damage in MMCs. They reported that with MMCs reinforced by particles with a size greater than 10 μm, the dominant damage mechanism is cracking of particle and that the particle-matrix interface appeared to have little effect on the overall damage fracture behaviour. Song et al.

[7] and Qin et al.[8] inferred through extensive experimentations that the ductility and fracture toughness of particle reinforced MMCs(PRMMCs) are affected adversely due to the presence of the hard and the brittle ceramic reinforcements. As a result of this, they claimed, many PRMMCs fracture with little warning that can be considered as detrimental to their structural applications. Llorea and Gonzalez[9] did a detailed analysis of failure and in the results they proposed that at the initial stages of plastic deformation the increase in load carried by the particles is mainly due to the progressive strain hardening of the surrounding matrix, which is relatively ductile. As the matrix strain hardening capacity is saturated, relaxation of stresses from fractured particles result in the stress transfer to nearby particles causing greater particle fracture. They further inferred that the final fracture of the composites takes place by a ductile mechanism involving the nucleation and growth of voids in the matrix, which ultimately coalesce around the broken particle. Weng et al. [10] suggested that saturation in strain hardening of narrow channels of matrix passing in between closely packed reinforcements raise the stress concentration around the reinforcements.

Liu et al. Inferred that matrix ductility is critical for the fracture toughness of PRMMCs. Also the mis-match between the CTE and elastic modulus between the two phases introduce residual stresses while fabricating the composite. In regions near the interface, strain hardening is caused by relaxation of these stresses. Even when a small external load is applied, strain hardening goes to a step ahead. Thus the limit of strain hardening is reached early, which falls short in case of application of a larger load, and eventually causes premature failure of the composite. The compressive residual stresses at the interface is an important parameter as it also aids in the decohesion of the interface. Hence they suggested that it is essential to have a knowledge of the residual stress level and its distribution on the composites during and after fabrication, when subjected to various temperatures and stress levels.

In stark contrast to this, Arsenault and Taya [11] and Wakashima et al. [12] have suggested that the coefficient of thermal expansion mismatch does not have much effect on the strength of the

material. They further put forth a point that in unreinforced alloys, no difference exists between compression and tension responses. This means the flow stresses in either case is almost similar. This, they attribute to the isotropic nature of the spherical reinforcements, which allow the residual stresses to cancel out.

In most of the cases, the matrix and the reinforcement are chemically not stable, resulting in slow diffusion of either of the constituent in to other. Such instability must be taken into account for slightest rise in processing or service temperature. Temporary elevation of temperature during processing is not much of concern as compared to prolonged exposure such as service. For such applications, thermo-mechanical compatibility, high temperature stability and the effect of the environment must be critically assessed.

Inter diffusion related phenomena, viz. dissolution of the reinforcement, formation of intermediate phases at the interface, contamination of the matrix by the fiber and vice versa, and coarsening of the reinforcements have adverse effects on the properties of the composites. Kopp et al[13]. have suggested that the understanding of inter diffusion between MMC components and their consequences is an indispensable tool in material selection for long term high temperature applications.

Chung et al. have deduced that the use of Cold Isostatic Pressing and Spark Sintering to make Aluminium alloy composites result in an increase in the hardness with the increase in the reinforcing content[14]. But Rajeev Kapoor, et al. suggest that at fixed strain, the hardening behavior is independent of the volume fraction and shape of the reinforcements, rather depends on the matrix properties[15].

Failure modes of MMCs are generally predominantly brittle in nature. Fracture in discontinuously reinforced composites can result from (a) debonding of particles from the matrix, (b) particle cracking leading to a matrix crack, (c) matrix cracking leading to composite failure. At room temperature under uniaxial loading, the predominant mode of failure observed in PRMMC is

particulate cracking followed by subsequent linkage in matrix[4][16][17].

2.3. Al-SiC MMC:

Research has shown that the addition of SiCp to Aluminium alloys would result in an increase of modulus, and may also be accompanied by an increase in yield stress depending upon the alloy composition, heat treatment, and manufacturing method. Furthermore it helps in increasing resistance to wear, corrosion and fatigue crack initiation as compared to the performance of the matrix alloy alone[18]. It has been reported that addition of SiC particulate reinforcement to Aluminium alloys usually lowers the fracture toughness[19]. However this drop in the fracture toughness has been found to be caused by the alterations in flow stress, fracture of SiC particulates[20], poor dispersion of SiC and a decrease in tensile ductility[21]. Other factors such as the volume fraction of the reinforcement, matrix alloy chemistry and processing variables have also been found to affect the composite character. But the interaction of these parameters are yet to be quantified to an extent that they can be deciphered.

Kapoor, et al. report that the addition of large ceramic particles to Aluminium alloys (esp. 6061) does not change the basic hardening mechanism of the matrix, however it is the formation of precipitates in the matrix that is responsible for the work-hardening behavior. Particle morphology and volume fraction were found to have little or no influence either. Work-hardening behavior rather appears to be microstructure dominated. The spherical reinforcements experience much lower stresses than their failure strengths. They merely serve as obstacles to the flow of the matrix or as deflectors of the cracks[15].

Weng, B.J. et al. Studied the micro-fracture mechanism of SiC-6061 Aluminium composite. They report that, for SiC/Al composites with 5 wt% - 10 wt% and 105 μm SiC, cracks initiated inside the SiC particles and penetrated into the nearby matrix. The crack proceeded from the cracked SiC particles. Ultimately a coalescence was observed among cracks emanating from

particles and matrix prior to the final fracture. For a similar composite with 10 wt% and 12 μm SiC, cracking initiated from the interface layer and was followed by severe deformation of the surrounding matrix material. A large amount of distortion was observed in the matrix. Cracks in the Aluminium matrix were blunted. Final fracture of the specimen was attributed to multiple fracturing and coalescence of the local cracks. For 5 wt% and 12 μm SiC, micro-fracture of the composite occurred predominantly by matrix cracking. Multiple slip bands were observed in the matrix. Cracking in discrete micro-constituents on the grain boundary was also observed and from there localized inter-granular fracture can be identified. Further localized deformation of the matrix proceeded, followed by linking-up of cracks until the fracture completed[22]. The results implied that particulates in the larger size range of 100 μm do not deflect the cracks, rather the cracks proceed through them. The smaller particulates effectively deflect the crack and sometimes inhibit crack propagation, thus facilitating a better mitigation technique. Rajeev Kapoor, et al. found from their experience with PRMMCs that larger particles have a higher probability of cracking. Llorca and Poza attribute this to the larger and more numerous surface flaws than smaller particles[23].

Sanjibani Pani reports that thermal shock at elevated temperature for samples sintered at relatively high temperature and thermal shock at low temperature for samples sintered at relatively low temperatures is less damaging for short-term use. But any type of thermal shock whether at low temperature or at high temperature is detrimental for long term use as it affects the strength properties or ductility or both[24].

2.4 Processing of Metal Matrix Composites:

Whether it is in the general knowledge of the community or not, composites are widely used in everyday use, but they lack accurate compositional balance. The reason is the complexity involved in fabrication of a critically designed composite.

Not all composites are fabricated using the same techniques. Each and every composite that

is designed for a specific purpose, needs to be fabricated in a route suitable for that composite only. Slight change in processing parameters may yield an altogether different set of properties. Despite this anomaly, there exist some generic routes which are used by many fabrication techniques more or less, in combinations.

2.4.1 Liquid state processing of MMCs:

Liquid state processing of MMCs find wide adoption because of the advantages associated in terms of lower costs of operation and the relative ease of fabricating complex intricate parts. The ease results from the requirement of fewer, simpler and known equipments and methods developed for the fabrication through liquid metallurgy route. However all these equipments and methods are specifically suited to unreinforced metals or alloys. More or less, all of them have some sort of undesirable effects which make impossible reproduction of dimensions to close tolerances. Further when they are used in the fabrication of reinforced materials, the effect is pronounced because of lack of precise control over process parameters. The liquid state processing is usually a high temperature process, which causes a number of chemical reactions at the interface of the reinforcement and the liquid metal, of which many reactions are undesirable. But in case of commercial production the advantages in terms of quantity (because of low cost and simplicity) outweighs the disadvantages (low quality), that's why majority of the MMC fabrication techniques are through liquid metallurgy route.

The generic term Liquid state processing refers to various methods employed to physically combine the matrix and the reinforcement. On such basis, the liquid state processes can be can be grouped into four major categories, viz.

1. Infiltration
2. Dispersion

3. Spraying
4. In-situ fabrication

2.4.1.1 Infiltration processes:

The process is to infiltrate liquid metal of the matrix material into a porous structure of the reinforcement either spontaneously or aided by external force. Practically a porous body of the reinforcing phase is held in a mold and molten metal is passed into it so that the voids and interstices are filled with the melt. Sometimes the melt does not wet the reinforcement spontaneously because of the wetting angle, and sometimes the capillary action prevents the entry of molten metal to the micro-pores; in such a case, external pressure is applied that helps in overcoming such opposing forces.

Variables involved in this process are the initial composition, morphology, volume fraction and temperature of the melt, the nature and magnitude of the assisting force applied to it.

2.4.1.1.1 No external force:

In cases when the metal spontaneously infiltrates the reinforcement preform or favours wetting, no external force is required to induce infiltration. Cermets, viz. Titanium Carbide reinforced steel or nickel base alloys have been produced by this method[25]. If in case spontaneous wetting is not favoured, the reinforcement may be pretreated to achieve wetting. Harington et al. developed one such process involving Ti-B, in which they deposited Ti and B onto the fibers through Chemical Vapour Deposition technique, to promote infiltration by Aluminium[26]. Another patented has been obtained by Lanxide Corporation who developed the PRIMEX process in which, Al-Mg alloy is made to infiltrate ceramic preform at high temperatures viz. 750 °C and 1050 °C in a nitrogen rich atmosphere. But the pitfall is that in absence of any steep driving force, the process tends to be very slow, to the tune of 25cm/hour only.

2.4.1.1.2 Vacuum driven infiltration:

To overcome the deficiency of the spontaneous process, a vacuum around the reinforcement is created which provides a sufficiently large pressure difference to drive the infiltration. Here instead of pouring of the melt from top, it is rather sucked upwards by the preform in the die cavity. The molten metal or its vapour reacts with the air present on top of the die to form solid MgO. Solidification results in further drop in pressure and this provides the necessary vacuum to drive the process.

2.4.1.1.3 Pressure driven infiltration:

This process is similar in principle to the vacuum driven infiltration except that, here the range of pressure is on the positive side and is applied externally. When the metal does not wet the preform (reinforcement), external force is applied to overcome the capillary forces which resist the entry of the melt. The assisting pressure may be applied by pneumatic means or with an actuating ram. Application by gas is done by forcing the of the metal into the preform of reinforcing phase by a suitable inert gas like Argon. The direct mechanical pressure is applied through the ram of a hydraulic press on the molten metal enclosed in a casing. In all the cases it is necessary to maintain the pressure throughout the solidification process.

The advantages reaped are increased processing speed, precise control over the chemical reactions, refined matrix microstructure, soundness of the product through feeding of shrinkages, and a port free matrix. However on the flip side, the application of pressure may induce preform deformation or even breakage during infiltration.

2.4.1.1.4 Other forces:

Alumina preforms are infiltrated by Al-Si alloys under low pressure with the assistance of vibrations[27]. Centrifugal casting methods have been adopted for producing a tubular reinforced

metal[28]. Electro-magnetic forces may also be used to circulate the molten metal to infiltrate the preform. Ultrasonic vibration may also be given to induce better wettability and remove voids.

2.4.1.2 Dispersion processes:

In this genre of processes the reinforcement is loosely incorporated in the metal matrix[29]. Since most of the metal reinforcements have poor wetting properties, a mechanical force is applied by means of stirring so that dispersion can be more uniform. This is the least expensive of all the processes employed in the primary production of MMCs, which can further be processed by casting or extrusion.

There are several variants of dispersion processes. The simplest one in use is the vortex method. In this method, a vortex is formed in the liquid metal and the reinforcement particles are added to the vortex[30]. A process for mixing SiC particulates in molten Aluminium under vacuum with the help of a specially designed impeller has been awarded a patent. The researchers Skibo and Schuster[31] claim that the process is advantageous in terms of limiting the incorporation of impurities, oxides or gases because of the vacuum and the reduced vortex. In a process called follow mixing, a rotating blade is progressively lowered in to an evacuated bed of particles covered with Molten Aluminium. Another variant of the process involves injection of particles below the surface of molten metal using a carrier gas.

The limitations of these processes are: poor control over the undesirable effects, viz. porosity as a result of gas entrapment during stirring, oxide inclusion, reactions between the matrix and the reinforcement due to prolonged exposure to high temperature, increased kinetics resulting in particle migration and clustering. Also this process is of little use when exact positioning of reinforcement is needed, viz. anisotropic composites consisting of long fibers and oriented reinforcements. Even if such a process is carried out, it requires secondary processing for improving the distribution which makes the total process lengthy and ultimately expensive.

2.4.1.3 Spray processes:

The matrix metal in molten form and the reinforcement in powder form is sprayed together on a substrate such that the reinforcement is randomly distributed interspersed with the matrix. Solidification completes on the substrate[29]. Parameters that affect the process are initial temperature, size distribution and velocity of the metal drops; velocity, temperature and feeding of the reinforcement. To atomize the molten metal, most atomizers use inert gases. The position of the nozzles from where the spray jet originates are of importance. Also important is the position, nature and temperature of the substrate. The reinforcing particles can be injected to the molten metal stream or somewhere midway between the liquid stream and the atomizer.

In this technique, due to the limited movement of the reinforcement allowed in the final structure, a very fine microstructure results with very less segregation. As the reinforcement and the matrix are have a very small duration of contact at high temperature, the reaction at the interface is minimized. This results in an excellent two-phase material which is thermodynamically metastable.

However this process is not without limitations. A high amount of residual porosity develops due to involvement of gases. This requires subsequent processing of the materials. Also owing to the use of high cost equipments and gases, this process tends to be costlier than the other options. The waste resulting from the out-of-target powder needs recycling and this raises the cost component.

2.4.1.4 In situ processes:

In situ processing methods encompass the techniques to develop the intended phase(s) in a blend by the use of some catalyst or some external trigger that create the reaction products at the intended sites. The term was first used for materials produced by solidification of poly-phase alloys. When these poly-phase alloys solidify directionally with a plane front, they may show very fine lamellar or rod like structure of β-phase in an α-phase matrix, the inter-phase spacing being a

function of growth rate of the lamella. Owing to excellent mixing of the precursors (usually in liquid state) this technique renders a very good distribution of the resulting reinforcement. However a major limitation is the low growth rates and problems resulting from the gradual coarsening of the structure of at high temperature because of the continued reaction. Researches are underway to produce reinforced inter-metallic alloys by controlled solidification and chemical reaction between the precursors.

Another way to produce in situ composite is to react molten metal with a gas. One such example is the production of Al_2O_3 / Al composite by oxidation of Aluminium. Another is injection of gases like CH_4 or Argon through a melt like Al-Cu-Ti, to produce wide range of carbide and nitride reinforced alloys.

The most significant advantage of this technique is the homogeneity of distribution[29]. But what mars the adoption is the limitation in choosing the system, preference of reinforcement orientation, control of the reaction kinetics which in turn means lack of control over the shape, size and orientation of the reinforcement.

The most promising area of its application is in the field of electronics. Along with it optical and structural applications have also been suggested.

2.4.2 Solid state processing of MMCs

Solid state processing of MMCs are generally used to obtain fine grained control over the composite microstructure and the reinforcement distribution. That is why it is the most preferred route for fabrication of discontinuously reinforced MMCs which enables one to obtain best possible results. Due to lack of freedom available in solid state, segregation effects and brittle reaction product formation are a bare minimum as against the liquid state processing route.

2.4.3 Primary solid state processing of discontinuously reinforced composites

Solid state processing of discontinuous reinforcements is preferred over liquid state processing because of the following reasons:

1. Lower temperature of operation, thereby avoiding any high temperature reaction
2. Ability to maintain segregation effect

The various techniques involved in solid state processing are:

1. Powder blending and consolidation
2. Mechanical alloying
3. Diffusion bonding and roll bonding
4. High-rate consolidation
5. Powder coating followed by solid-state consolidation

2.4.3.1 Powder blending and consolidation

The matrix material and the reinforcing material are mixed uniformly to obtain a homogenized mix. A green compact is obtained by suitable pressing, viz. uniaxial pressing or cold isostatic pressing. This green compact is sintered at a suitable temperature under controlled atmosphere to regulate the various reactions occurring. The choice of temperature and atmosphere depends upon the properties intended to improve. Sintering may be done either in the solid state or in the liquid state. Solid state is preferred when the segregation is required to be retained unaltered. Though in certain cases the temperature may be raised to just above the matrix solidus temperature to enable the interface to form properly, to minimize the deformation stresses and to avoid any particulate damage that may otherwise occur in severe shear conditions during processing.

In liquid state sintering, external pressure is seldom required because of the natural tendency of the low-melting phase to pull the solid particles together due to surface tension effects. The ideal

condition to impart appropriate bonding and the creation of a well-defined interface through liquid state sintering is that the high melting phase should be slightly soluble in the low melting phase.

Sometimes the green compacts are thoroughly out-gassed and forged or extruded to obtain the composite. Alternately the green compacts or the powder blend may also be subjected to hot isostatic pressing after complete out-gassing.

2.4.3.2 Mechanical alloying:

This is a solid state mixing route. Powder mix in pre-calculated ratios are violently mixed in a planetary ball mill. The high energy impact continuously fragments and cold-welds the powder particles thus exposing fresh internal surface continuously resulting in fine grained structures in the end. The almost homogenized mixture can be subsequently processed through hot pressing, extrusion,etc. to consolidate the composite.

2.4.3.3 Diffusion bonding or roll bonding:

Diffusion bonding of materials in the solid state is a process for making a monolithic joint through the formation of bonds at atomic level, as a result of closure of the mating surfaces due to the local plastic deformation at elevated temperature which aids inter-diffusion at the surface layers of the materials being joined[32]. Application of pressure plays the key role in this process where in the principal mechanism is inter-diffusion of atoms across the interface. Requirement of pressure and its holding time for the consolidation to complete can be determined form prior knowledge of flow stress of the matrix to actuate its flow between the fibers and across the interfaces of the foils brought in contact. Laboratory scale diffusion bonding of most metals is carried out in vacuum or in an inert atmosphere to prevent formation of reaction products which lower the mechanical strength.

The composite can be prepared by processing metallic foils and fibers/ particulates through roll bonding or co-extrusion processes. It can be done through either of the two below-mentioned ways. Powder blends packed and evacuated in a container are subjected to the consolidation methods through roll bonding. Alternatively, laminated composites are produced by high temperature roll bonding operations starting from either of the alloys or the individual metals. During the process, a strong interface is created by both, surface deformation and diffusion, causing asperity deformation and inter-diffusion.

2.4.3.4 High rate consolidation:

This method of consolidation is highly suitable for the rapidly solidifying and hard to deform metals. Usually the dynamic action of a shock wave is employed to produce a monolith from powdered starting materials. The die used in this process is a singular cavity carved on to a solid with the pressure plate being backed by reflectors which rebound the shock wave for enhanced consolidating effect. For batch processing, many die cavities may be carved on to a single block.

The principle of operation is that, frictional heating generates a lot of heat at the interface which is extracted by the cooler interior, thus causing rapid solidification. As because this is a forced consolidation, large amount of dislocation are retained inside. Though this results in increased strength, but at the expense of ductility. Sometimes this process results in cracked products.

2.4.3.5 Powder coating followed by solid state consolidation:

The reinforcement powder is coated with some metallic/ceramic material by electrochemical, thermo-chemical, vapor deposition methods. This aids in boosting the properties of the reinforcement. Because of the thickness of the coating around the reinforcement, the inter-particle spacing gets increased. This is a distinct advantage, as most of the composite failures are

attributed to lowered mechanical strength caused by very narrow inter particle distance or at times the contact of reinforcing particles. However the coating technologies in use are not vary advance and hence the requirements to apply coatings of contamination free uniformly thick are seldom met with success. Hence very few fabricators resort to this sort of coating application.

Blending of the metal matrix composite mix is usually carried out in V-blenders or in other agitating devices such as planetary ball mill, etc. Atomized pre-alloyed powder of the matrix material (alloy or elemental mix) is mixed with suitable proportion of reinforcement powder or whisker. This mixture is placed in a suitable mixing equipment and an inert atmosphere is maintained to prevent formation of reaction product. It has been observed that agglomeration of reinforcing particles occur if the size difference between the matrix powder and the reinforcement powder is very large. This can be overcome by the addition of a suitable surfactant, which creates a repulsive force and hence improves the distribution of the reinforcement. But this practice is usually avoided, because addition of surfactant may result in contamination and the mechanical properties at the end may be different from that expected. Hence the most preferred route is blending in a V-blender with an inert atmosphere without the addition of any surfactant.

The usual powder consolidation technique is a typical solid-state one. In some exceptional cases liquid-state process is adopted, but it is limited to high-temperature composites. The reason for avoiding liquid-state sintering is that the freedom available with liquid phase of matrix causes the segregation of reinforcement particles, the high temperature promotes formation of inter-metallic formation and grain boundary separation. Wherever it is not possible to avoid liquid state sintering, and at the same time segregation is to be averted, ultrasonic agitator is used in the liquid slurry. Hence the consolidation is done by hot pressing or hot isostatic pressing at temperatures as high as to have the softest state of the matrix but without giving freedom to the reinforcement to segregate. Most discontinuously reinforced composites are subjected to deformation processing which improves their microstructure and mechanical properties. This is also particularly helpful to

render a shape.

2.4.4 Deformation processing of metal matrix composites:

Deformation processing is the secondary processing of the particulate reinforced composite. Composite materials are less ductile and more prone to internal damage (voiding and particle fracture) than their unreinforced counterparts. Hence secondary deformation working is done at around the hot working temperature range where the matrix material does not work harden[33]. This leads to reduction or elimination of porosity and improves bonding. These factors separately or collectively aid in improvement of the mechanical properties of the MMCs. The secondary deformation processing may include extrusion, rolling, forging of MMCs.

2.4.4.1 Extrusion of consolidated MMCs:

Extrusion is the most common secondary processing of MMCs because of involvement of a high amount of hydrostatic pressure that prevents internal damage from occurring easily. It is carried out at a high strain rate and a temperature that ensures high strain rate sensitivity. It involves dislocation and creep deformation of the matrix. The high strain rate breaks the oxide layer on the Aluminium particles. But a very high strain rate is likely to crack the reinforcement thereby being detrimental to the mechanical strength. The strain rate is so optimized that along with the maximum possible rate the most uniform flow of the matrix is obtained. The advantage of extrusion is that, apart from improving the homogeneity of the composite, it can produce long net-shaped products.

Presence of 15-25% of non-deformable particulates as in Al-SiC MMCs may cause fracture of the reinforcing phase during extension. It may also cause “Christmas tree” effect, the ragging of the edges of extruded components[34]. To avoid this condition, the extrusion die is tapered keeping in mind the shape of the dead metal zone, or the extrusion is done in high pressure fluid atmosphere

that drastically limits the friction and promotes a better distribution of pressure. Gunashekhar et al. have shown that the use of streamlined dies can produce many complex-section shapes without cracks which would be impossible with the use of conventional extrusion die shapes[35]. Several microstructural changes occur during extrusion. These are alignment of particles along the extrusion axis, particle fracture, refinement and recrystallization of matrix grains.

2.4.4.2 Rolling of consolidated MMCs:

If sheets or plate like structure are required then rolling is done (usually after extrusion). As because the sides are unconstrained, and most of the composites are less ductile, edge cracking is very common in rolling. Therefore rolling to less than 40mm thick sections often leads to a significant amount of cracked material which needs to be scrapped ultimately. Cold rolls can also contribute to cracking because of the high temperature gradient generated during rolling to thin sections. Hence rolling of discontinuously reinforced MMCs is most successful in the temperature range of $0.5T_m$ (melting temperature) at low roll speeds.

If it is not desirable to raise the temperature to $0.5T_m$, then warm isothermal rolling using smaller passes and a large roll diameter can produce sheets out of the discontinuously reinforced MMCs with a minimum of edge-cracking. Alternately at lower temperatures, reduction per pass must be made very small and intermediate annealing steps should be introduced to limit the stress generated.

2.4.4.3 Forging of consolidated MMCs:

Many uses of MMCs depend upon forged products. Automotive connecting rods, missile components, navigational systems, structures for space applications, etc. all use forged parts. But the pitfall is that forging of these products is often limited by cracking in the outer surface caused during the forging operation itself. It has been observed that the work piece in a closed die forging

operation may develop incipient cracks at an intermediate stage of forging but may no longer be visible at the end of the operation. This type of crack is detrimental for fatigue loading and hence the knowledge of forging limit for the appearance of outer surface cracks is essential.

2.5 A birds eye-view of the literature surveyed:

Sl no	Name of Author(s)	Conclusive findings
1	Ashby	New materials with tailored properties can be made from pre-existing materials. As there is freedom to choose the ingredients, there is indirect freedom to choose the required properties and derive intended results.
2	Srivatsan	Discontinuously reinforced Aluminium alloy composites find their use in weight-sensitive and stiffness critical component. The discontinuous reinforcement in Aluminium alloy metal matrix develops properties not attainable by other means. Non-uniform size and dispersion of the reinforcing phase caused the particles to crack at low values of applied stress. Incresing the SiCp volume fraction resulted in higher fatigue strength For a given volume fraction of the SiCp, fracture morphology was observed to be essentially similar at different cyclic stress amplitude With an increase in SiCp contents in the metal matrix, fractures of the composite were dominated by particulate cracking and decohesion at the interface.
3	Beatrice Lay	Effect of a soft interface between the particle and the matrix shows that the stress concentration towards the surface of the reinforcement diminishes substantially. The precipitate-free zone around the reinforcement appears to limit the surface cracking of the SiC and, consequently, to increase the ultimate strength of the composite.

4	Lloyd et al. Whitehouse D.J., Ribes et al.,	For MMCs reinforced by particles with a size greater than 10 μm, the dominant damage mechanism is cracking of particle and the particle-matrix interface appeared to have little effect on the overall damage fracture behaviour
5	Song et al. Qin et al.	The presence of hard and brittle ceramic reinforcements adversely affect the ductility and fracture toughness of particle reinforced metal matrix composites.
6	Alcan corp. Brochure	Enhancement of mechanical properties of reinforced metals with simultaneous degradation of ductility and fracture toughness
7	Jagnes, et al.	The matrix is responsible to keep the reinforcement in place and is responsible for transfer of the loads to the reinforcement.
8	L lorea, et al.	Final fracture of the composite takes place by a ductile mechanism involving growth of voids in the matrix. Finally these voids tend to coalesce and form larger voids that originate around larger particles.
9	Liu, et al..	Matrix ductility is critical for the fracture toughness of PRMMCs. Mismatch in the CTE of the matrix and the reinforcement gives rise to residual stresses in the amtrix during fabrication of the composite itself.
10	Kopp, et al.	Interdiffusion of the components of MMCs helps in selecting the components for composite processing for long-term high temperature applications.
11	Foo K.S., et al.	Liquid phase process resulted in strong bonding between the reinforcement and the matrix. No Al_4C_3 were formed but other intermetallics viz. Mg_2Si and $FeSiAl_3$ were observed.
12	Jogi B.F, et al.	Plastic deformation of MMC under compressive loading proceeds by two mechanisms-"grain deformation" and "boundaries slip"- according to the bonding strength among the different powder particles. Characteristic behaviour of MMC can be understood according to the size and content of the reinforcement.

13	Showaiter N, et al.	Sintering shows formation of both transient liquid phase at temperatures between ~320°C and 550°C and persistent liquid phae above solidus temperature at ~590°C. Hence it is believed that sintering mechanism is via the formation of both transient and persistent liquid phases. Sintering under vacuum resulted in higher porosity, where as sintering under pure nitrogen environment provided higher densities of ~97-99% TD with sintering aids.
14	Kapoor R, et al.	Addition of large ceramic particles to Aluminium alloys does not change the basic hardening mechanism of the matrix, rather the formation of precipitates in the matrix significantly influences the work-hardening behaviour. Particle morphology and volume fraction have little or no influence on the work-hardening behaviour which appears to be matrix microstructure dominated. In compression, the stresses experienced by particles are much lower than their failure strengths; hence the particles merely act as obstacles to the flow of the matrix. Larger particles show greater propensity to crack than smaller particles owing to the higher probability of larger particles having a critical sized defect.
15	Nieh, Wadsworth	It is possible to attain superplastic like flow and tensile elongation of up to 300% in a 20vol% SiCw composite at 525 °C using a high strain rate of 3.3×10^{-1} s^{-1} . Such a high strain rate superplasticity provides a practical route for the plastic forming of SICw/ Al composites.
16	Mabuchi M, et al.	Partial melting temperature of the SiCw20vol%/Al 6061 composite is 579.5°C and that of 6061 Al alloy is 589.4°C respectively. The possible reason of having a lower melting temperature is that there more interfaces exist in the SICw reinforced composite. Mg and Cu segregate at the interface which can reduce the solidus temperature at the interface.

17	Gnjidić Ž. et al.	The presence of SIC particulates causes the aging process to be accelerated due to the increased dislocation densitiy, that provides more sites for the nucleation of precipitates.
18	Sugimura, et al., Liwa, et al., Davidson, et al. Manoharan, et al.	The factors limiting the use of the DRA composites are the low value of fracture toughness and poor tensile ductility compared to the unreinforced alloys.
19	East W.F. Demeis R.	They have quantified the increase in strength and stiffness of SiC particulate reinforced ductile Aluminium alloys. They maintain that SiCp is the most preferred reinforcement for Aluminium alloys.
20	Nair, et al. Mc Danels D.L.	They conclude that the increase in strength of the SiCp reinforced Alminium alloy composites became more pronounced at elevated temperatures
21	Will T.C.	100% improvement in elastic modulus was observed by incorporation of 40% SiCp reinforcement in an Aluminium alloy compared to the unreinforced alloy.
22	Davis, et al.	The mismatch in the CTE between the SiCp and Aluminium alloy matrix gives rise to a high density of dislocation at and near the matrix-reinforcement interface.
23	Sinclair, et al..	PRAMMCs have attractive material characteristics. Fabrication of continuous fiber, short fiber or whisker reinforced Aluminium matrix composite is not cost friendly.
24	Srivatsan, et al.	Al-SiCp MMCs develop a higher strength because of the residual stress as a result of CTE mismatch and the considered plastic flow of the matrix due to the triaxiality of the ductile Aluminium as a result of the presence of the dispersed reinforcement.
25	Tham, et al.	When limited to a thin layer, chamical reactions between the matrix and the reinforcement promote intimate interfacial contact and therefore the strength of the metal-ceramic bond. The strain to failure in a composite is primarily governed by the volume fraction of the matrix phase that flows physically durong deformation.

26	Iseui, et al.	SiC particles may react with liquid Aluminium to form Al_4C_3 and Si, during processing itself.
27	Arsenault and Taya	CTE has little or no effect on the compressive strength of the final composite.

Table no. 1: A bird's eye view of the literature surveyed

2.6 Conclusion:

The literature survey has briefly detailed the work of various works till date. It covers their experimentation, characterization, important observations, conclusive findings.

CHAPTER 3
EXPERIMENTAL

3.1 Introduction:

This chapter describes the experimental procedure as adopted in the present project work. The equipment / instruments used for the various experiments in this work are listed in a tabular form depicting their specific contextual uses, their specification and particulars.

A detailed report is also provided on the raw materials used for fabrication of the test specimen and the characterization of the raw material used for fabrication. Details of each procedural step adopted for the fabrication of the test specimen, their heat treatment profile, the methods of mechanical testing carried out, the generation of the micrographs through Scanning Electron Microscopy(SEM) has been furnished.

For the sake of clarity and brevity, photographs of equipments / instruments that have been used in this work are also inserted.

3.2 Equipments / instruments used:

Sl. no.	Instrument/ Equipment	Specification	Use in the present investigation
1	Surface milling machine	Make : ----,Russia	For producing chips from the Cast Aluminium alloy billet.
2	Muffle furnace	Make: Wild Barfield Model: HT25 Max. temp.: 1550 ºC	For pre-treatment of the SiC particulates at 700ºC to impart a coat of SiO_2
2	Planetary ball mill	Model: Pulverisette-5 Make: Fritsch, Germany Medium: Chrome steel balls(9.5 mm) , stainless steel balls (10 mm)	(i)For preparing powders form the Aluminium alloy chips (ii) For mixing of Aluminium alloy powder and SiCp thoroughly to obtain an uniform mixture
3	X-ray diffractometer	Make: Philips Model: Analytical X-Ray X'Pert-MPD system Type: PW3040 2θ range: 0º-160º	For characterization of the Aluminium alloy at various stages to ascertain the composition and monitor the level of contamination.

4	Cold-uniaxial hydraulic press	Make: SoilLab Type: Hydraulic Max. Load: 20 tons	For preparing the green test specimen by compaction of the alloy-reinforcement powder mix.
5	Cold-isostatic press	Make: 1999 Type: Hydraulic Max. pressure: 400 MPa Work temp.: Ambient canister size: 11 ltr.	For cold-isostatic pressing of the green test specimen as obtained from the cold uniaxial press.
7	DSC-TG		For finding the reactions
8	Dilatometer	Make: NETZSCH Model: DIL402C	For finding the strain of the specimen when exposed to the sintering temperatures.
9	High temperature Horizontal tubular furnace	Make: Naskar & Co. Type: Vacuum and Controlled atmosphere Max. temp.: 1750 ºC Dimensions: $Ó_{out}$-85mm $Ó_{in}$-75mm	Used for sintering the samples, obtained after cold-isostatic pressing, in a Nitrogen atmosphere at various sintering temperatures with different lengths of time.
10	Electric Oven	Range: 30ºC-300ºC	For exposing the samples to a thermal shock at +80 ºC, an elevated temperature.
11	Cryogenic chamber	Make: S.D. Scientific Industries Ultra low chamber Range: +50 ºC to -80 ºC	For exposing the samples to a thermal shock at a sub-ambient temperature, -80ºC
12	Instron-1195	Make: Instron Ltd. Model: 1195 Range: 0.1N - 100kN	For mechanical testing of the sintered specimen (Compression test)
13	Scanning Electron Microscope	Make: JEOL Type: JSM-6480LV	For obtaining the micrographs of the fractured surfaces of the test samples subjected to compression test.

Figure no. 1: Surface milling machine

Figure no. 2: Muffle furnace

Figure no. 3:
Planetary ball mill

Figure no. 4:
X-Ray Diffractometer

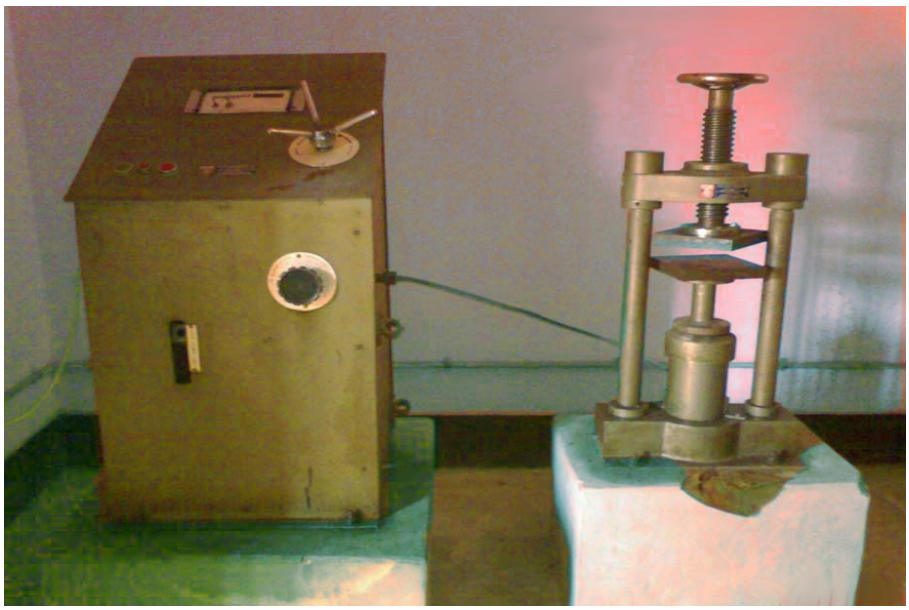


Figure no. 5: Cold uni-axial press

Figure no. 6: Cold-isostatic press

Figure no. 7: High-temperature Tubular furnace

Figure no. 8: Electric Oven

Figure no. 9: Ultra low Chamber

Figure no. 10: Instron (Model:1195)

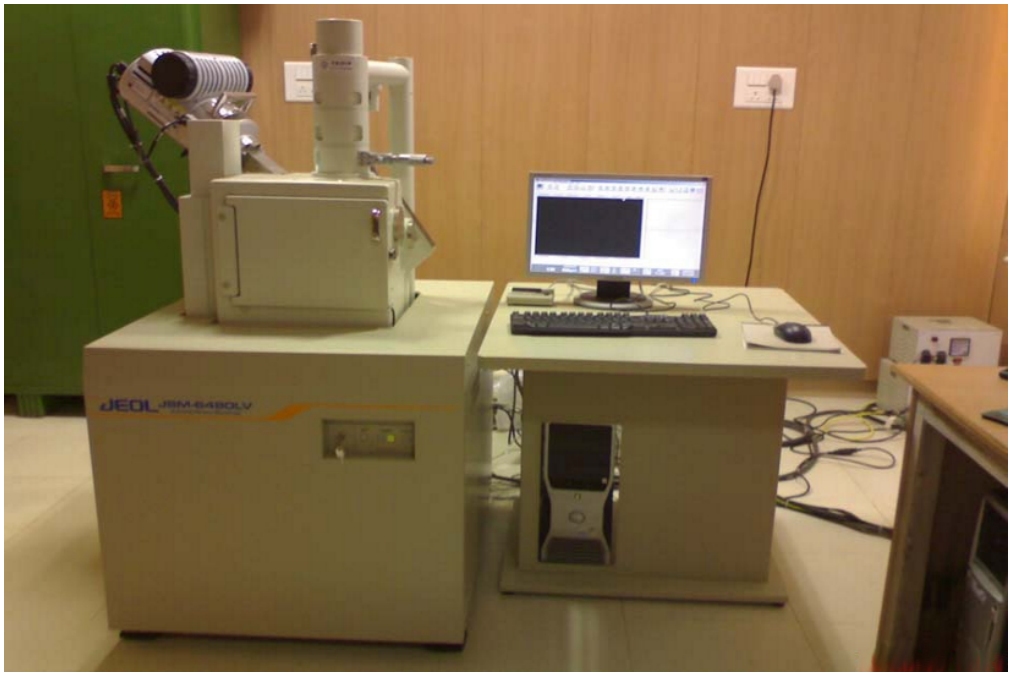


Figure no. 11: Scanning Electron Microscope

3.3 Selection and characterization of raw materials:

Commercial grade Aluminium alloy 6061 billets were obtained from PES Institute of Technology, which in turn had acquired it from Fenfe Metallurgicals, Bengaluru. The SiC particulates were obtained from the market.

The specifications/composition obtained are presented below.

3.3.1 Aluminium Alloy:

Si: 0.4-0.8 %max

Fe: 0.7 %max

Cu: 0.15-0.40 %

Mn: 0.15 %max

Mg: 0.8-1.2 %

Cr: 0.4-0.35 %

Zn: 0.25 %max

Ti: 0.15 %max

3.3.2 SiC particulates:

Manufacturer: Alfa Aesar

Assay: 99% (metal basis)

Particle size: 33.62 μm.

3.3.3 Pre-treatment of SiC particulates:

If SiC is used as a reinforcement in an Al alloy matrix containing less than 7% Si, then the Al from the matrix migrates to the SiC reinforcement and reacts with it as per the following reaction:

$$4Al + 3SiC \leftrightarrow Al_4C_3 + 3Si$$ [Azom internet site]

Formation of Al_4C_3 is detrimental to the mechanical property of the composite. Hence to prevent the reaction, SiC and Al need to be isolated. To achieve this, the following treatment is resorted to.

As prescribed by T. Sritharan et al., the SiC particulates are heated to a temperature of 700°C and held at that temperature for 120 minutes in a muffle furnace in presence of air. This deposits a thin coat of 2µm thick SiO_2 over the SiCp. This barrier coating prevents the migration of Al into SiCp and hence prevents the reaction product from forming.

3.4 Fabrication of the green test specimen:

The MMC test specimen are fabricated by powder metallurgy route using ball mill mixing and solid state sintering.

3.4.1 Mixing of the powders:

90% Aluminium powder and 10% SiCp by weight are mixed for fabricating the composite. Mixing is carried out in a planetary ball mill with chrome steel vial and stainless steel and chrome steel balls. Two of vials were employed with 60 balls in each. The inner diameter of the vials were 75mm and the average diameter of the balls were 10mm for the stainless steel ball and 9.5 mm for the chrome steel balls. Referring to previous works, the speed of rotation was chosen to be 300rpm[24].

3.4.1.1 Optimization of mixing time:

Mixing was carried out for a period of 30minutes and the the machine was stopped for a period of 30 minutes to cool down the vial. This stop and go sequence was repeated until 6 hours of cumulative mixing time

3.4.2 Compaction of the powder mix:

About 5.6 grams of the powder mix was taken adopting a method of coning and quartering for compaction in a cold uniaxial press in a metallic die-punch arrangement.

3.4.2.1 Cold Uniaxial pressing:

The above mentioned powder mix was pressed in the Cold uniaxial pressing machine at an incident load of 4 tonnes. A stainless steel die of 11.8 mm internal diameter was used for this purpose. To allow the powder to flow freely and to prevent the specimen from sticking on to the walls, stearic acid was used as a lubricant that was applied to the walls of the die and punch.

3.4.2.2 Cold isostatic pressing:

The green test specimen were now subjected to cold isostatic pressing. The samples were kept in latex tube; the tubes were evacuated with the help of a vacuum pump and sealed. All the samples were then kept in an oil bath in the cold isostatic press chamber. The samples were subjected to a pressure of 250 MPa and held at that pressure for 15 minutes to allow proper penetration.

3.4.3 Sintering of the green pellets:

Green pellets obtained after cold isostatic pressing were subjected to sintering in a high temperature tubular furnace. Sintering was done in the solid state only. Sintering temperature and the time of holding were varied. Previous workers had shown that melting of the composite was occurring in a temperature range of 582-652 ºC. A confirmatory DSC test on the specimen showed that partial melting started as early as 450 ºC . The Dilatometry results showed that at around 500 ºC the instantaneous strain percentage was zero. Around 540 ºC it attained negative values. This meant the temperature range around 500 - 540 ºC was an ideal zone for investigating the properties

of the composite. Hence, the sintering temperatures were chosen to be 500 ºC, 520 ºC, 540 ºC, 560 ºC respectively. With each temperature, the time of holding was varied as 1 hour, 2 hours and 3 hours. For each combination of time and temperature, different batches of samples were treated.

Each batch of sample comprised of 3 specimen. Each batch was placed in the tubular furnace. The tube was sealed and evacuated to 10^{-4} mbar. N_2 gas was passed through the tubular furnace for the entire duration of sintering to prevent oxidation of Aluminium and to induce the formation of AlN. Heating rate was maintained at 5 ºC/minute.

3.4.3.1 Nomenclature of the test specimen:

Temeperature	Time	Thermal shock		
		No shock	+80ºC	-80ºC
500 ºC	1	500-1-0	500-1-1	500-1-2
	2	500-2-0	500-2-1	500-2-2
	3	500-3-0	500-3-1	500-3-2
520 ºC	1	520-1-0	520-1-1	520-1-2
	2	520-2-0	520-2-1	520-2-2
	3	520-3-0	520-3-1	520-3-2
540 ºC	1	540-1-0	540-1-1	540-1-2
	2	540-2-0	540-2-1	540-2-2
	3	540-3-0	540-3-1	540-3-2
560 ºC	1	560-1-0	560-1-1	560-1-2
	2	560-2-0	560-2-1	560-2-2
	3	560-3-0	560-3-1	560-3-2

Table no. 3: Nomenclature of the test specimen

3.5 Exposure of the test specimen to thermal shock:

For exposing the samples to thermal shock, the specimen were divided into three groups. Each group was formed by taking one sample for each combination of sintering temperature and sintering time. One group was exposed to +80 ºC in an electric oven, one to -80 ºC in a cryogenic chamber and another group was kept at room temperature. Thermal shock was given for a duration of 1 hour. After the samples completed one hour at the shock temperature, they were removed and kept in a thermally insulated flask. All samples were then subjected to mechanical testing in an Instron (model 1195).

3.6 Determination of the compressive strength:

Compression test was done on an Instron 1195 universal testing machine. The samples were prepared as per the ASTM E9 (extended) standard for compression test of hard metals. The cylindrical specimen were compressed at a crosshead velocity of 1mm per minute. The readings were available from a calibrated automated loadcell.

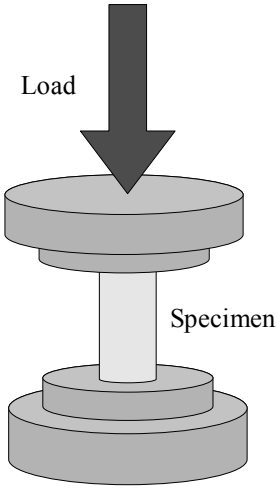


Figure no. 12:
Schematic diagram of the compression test arrangement

3.7 Scanning electron microscopy:

The fractured test pieces were examined with the help of a Scanning Electron Microscope (JSM-6480LV). The fractured surfaces were studied for modes of fracture, the failure of the interface, failure of the matrix, failure of the reinforcement, etc.

3.8 Conclusion:

This chapter provides the details of the testing equipments, processes and procedures adopted in the present work.

Fig. no. 13: Sintered specimen before fracture

Fig. no. 14: Sintered specimen after fracture

CHAPTER 4
RESULT & DISCUSSIONS

4.1 Introduction:

This chapter houses the experimental results. The data are plotted and also presented in the format of bar-diagrams. The experimental data are examined and analyzed in great details. The variation of experimental findings are explained on the basis of related work in the field and the theory available.

Also an attempt is made to analyze the mode of failure; for this purpose SEM micrographs of the broken specimen surfaces are exhaustively studied and analyzed. On the basis of the analysis, the likely mode of failure has been predicted.

4.2 Assessment and evaluation of Mechanical testing:

4.2.1 Load at rupture under thermal shock

Sl no	Sintering temp ºC	Time at temperatures	Load at rupture under different thermal exposures (MPa)		
			No thermal shock	Exposure to +80 ºC	Exposure to -80 ºC
1	500	1	15.12	17.07	17.02
		2	15.69	17.45	16.75
		3	16.72	18.98	16.70
2	520	1	16.74	18.93	16.12
		2	18.14	18.79	16.42
		3	19.50	17.85	17.46
3	540	1	19.60	20.13	17.74
		2	19.65	21.43	19.05
		3	20.02	22.81	12.64
4	560	1	20.08	18.76	18.12
		2	20.20	19.63	17.06
		3	20.49	17.90	17.79

Table no. 4: Table showing load values at rupture for samples at various sintering temperatures, holding times and thermal shocks given.

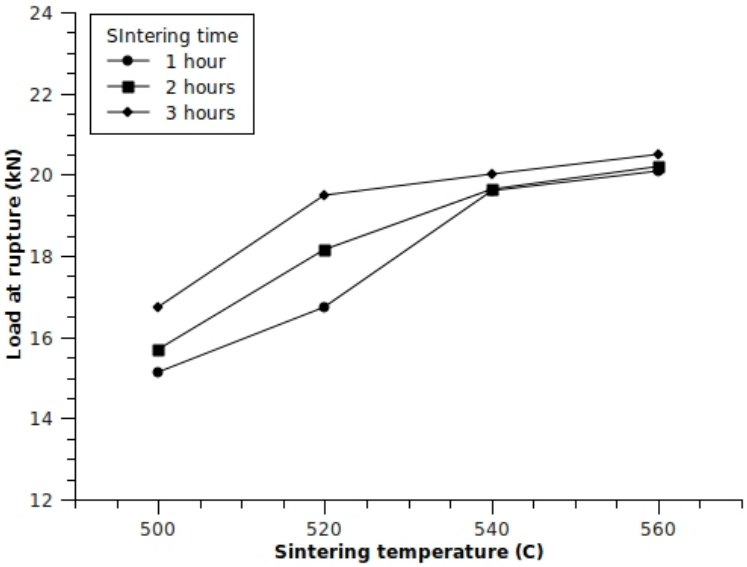


Fig no. 15:
Load at rupture Vs. Sintering temperature as a function of Time at temperature for samples not subjected to any thermal shock.

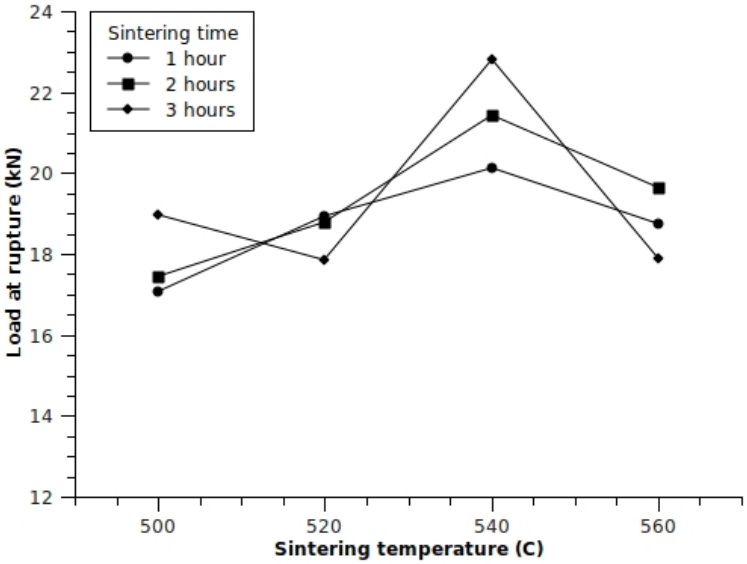


Fig no. 16:
Load at rupture Vs. Sintering temperature as a function of Time at temperature for samples subjected to thermal shock at high temperature of +80 ºC.

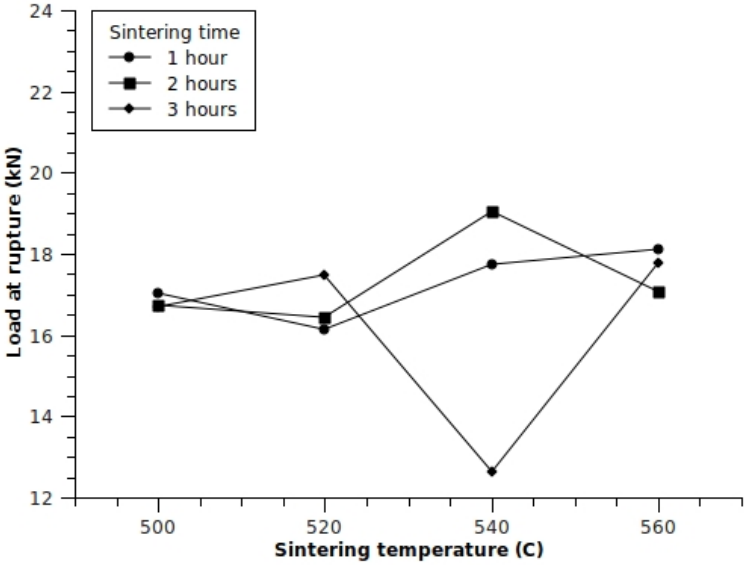


Fig no. 17:
Load at rupture Vs. Sintering temperature as a function of Time at temperature for samples subjected to thermal shock at high temperature of -80 ºC.

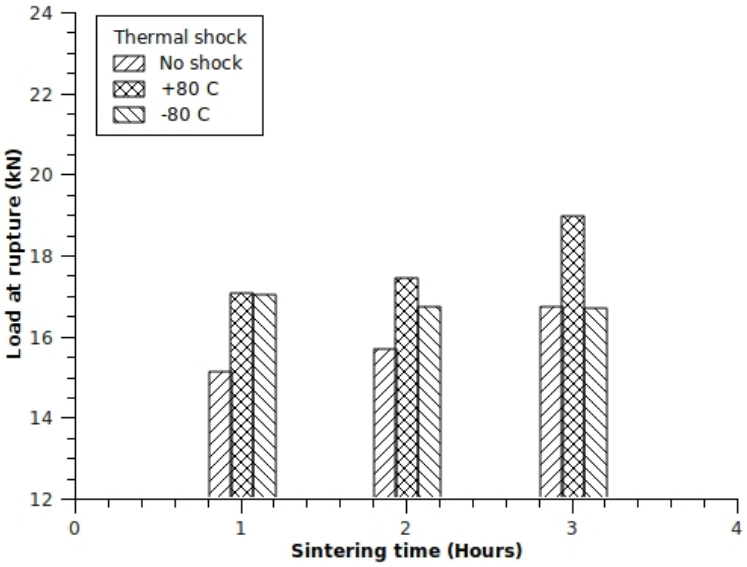


Fig no. 18:
Load at rupture Vs. Time at sintering temperature (500 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

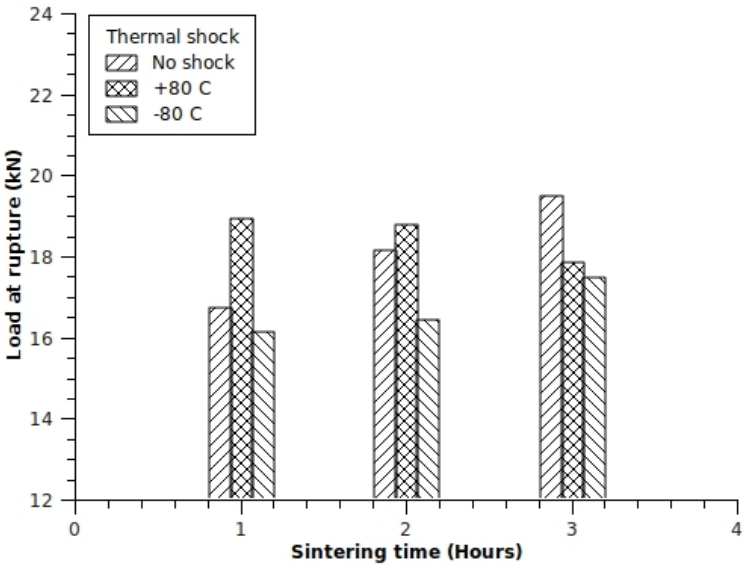


Fig no. 19:
Load at rupture Vs. Time at sintering temperature (520 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

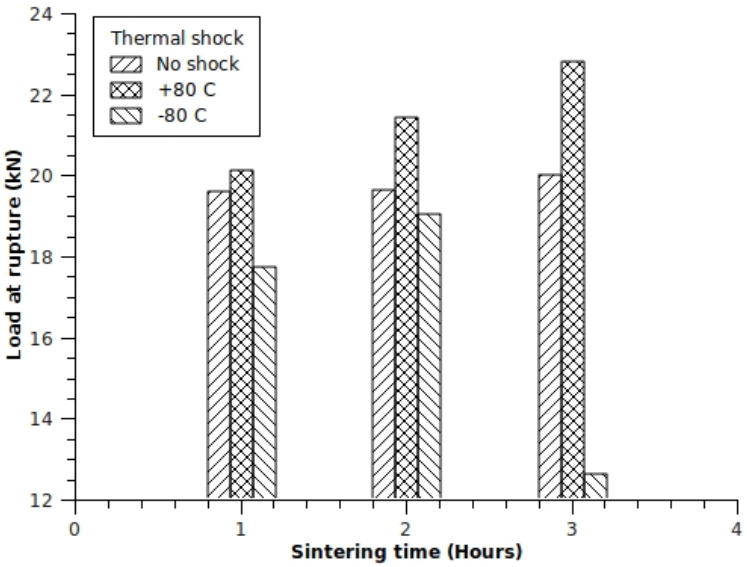


Fig no. 20:
Load at rupture Vs. Time at sintering temperature (540 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

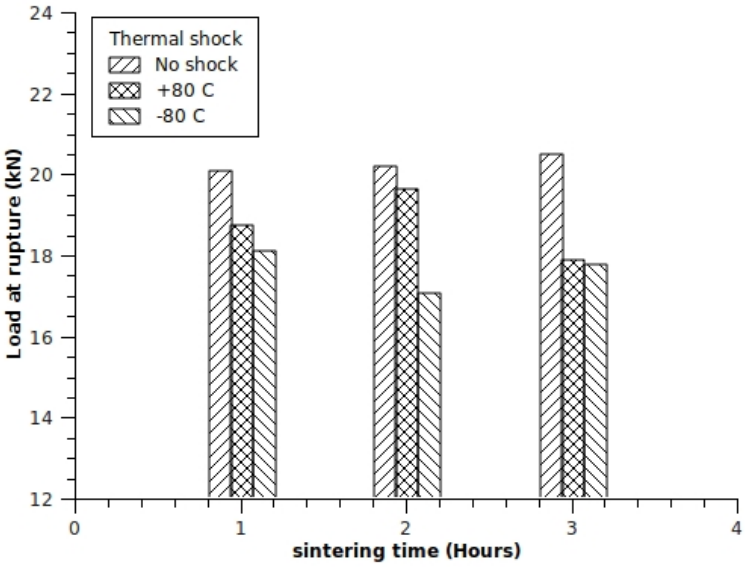


Fig no. 21:
Load at rupture Vs. Time at sintering temperature (560 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

Figure no. 15 shows the variation of load at rupture as a function of different sintering temperatures when held at the sintering temperature for various lengths of time. Fig. no. 16 and 17 represent the same data when the samples are exposed to thermal shocks of +80 ºC and -80 ºC respectively.

Fig. no. 18 through 20 represent the bar diagrams for load at rupture as a function of holding time at the respective sintering temperatures for samples without any exposure to thermal shock of +80 ºC and -80 ºC. The time at temperature is varied from 1 hour to 3 hours at steps of 1 hour interval and the sintering temperature is varied from 500 ºC to 560 ºC at steps of 20 ºC each.

The data and the figures reveal that for the highest temperature of sintering (560 ºC), the stress at rupture increases with the increase in the time at temperature for the samples not exposed to thermal shock. This can be attributed to the inter-atomic diffusion between the matrix and the reinforcement, which is temperature as well as time dependent.

On a further examination, the data reveal that for the samples without any exposure to thermal shock at all temperatures of sintering, longer holding time always enhances the load at rupture and that with the increase in sintering temperature, the load at rupture at respective holding times are higher for longer holding times.

The samples sintered at 560 ºC and held at the temperature for 1 hour exhibit a higher value of the load at rupture compared to that held at the temperature for 3 hours, when the thermal shock is due to an exposure to +80 ºC. This trend continues for the samples at the same high temperature of sintering with a different holding times when the thermal shock is due to the exposure to -80ºC. However, the relative values of load at rupture with shock due to -80 ºC are lower than that due to shock at +80 ºC. Thus, it can be concluded that the thermal shocks at +80 ºC and -80 ºC are less damaging for those samples which are sintered at relatively higher temperatures but held at the sintering temperatures for shorter lengths of time and that this trend is reversed for samples sintered at higher temperatures with a longer holding time. It can also be concluded that for all these cases of samples sintered at a higher temperature, the thermal shock due to an exposure to sub-ambient temperature (-80 ºC) is more damaging than the same caused by an exposure to an elevated temperature (+80 ºC).

The load at rupture for sintering temperature of 540 ºC and 520 ºC both reveal that for holding time of 1 hour, the value assumes a higher dimension when exposed to +80 ºC than that at -80 ºC. It is further revealed that at both the temperatures, the load at rupture in all cases of holding time are less than that compared for representative holding times at the higher temperature of 560 ºC. The only exception is 540 ºC with 2 hours holding time, which may be due to an experimental error.

When the sintering temperature is much below the melting point of the matrix, the resistance to thermal shock is increased with the increase in the holding time at the sintering temperature for a thermal shock due to exposure to +80 ºC. This is revealed from the fact that the load at rupture values are increased for the samples sintered at 500 ºC when the holding time is increased from 1 hour to 3 hours with the thermal shock caused due to an exposure to +80 ºC.

This trend is reversed when the thermal shock is due to an exposure to -80 ºC. The samples sintered at 500 ºC show an increase in the load at rupture values with the decrease in the time of holding at the sintering temperature. It may be recorded, however, that even the highest values of load at rupture at lower sintering temperature of 500 ºC is lower than the lowest of those at higher temperatures.

It is further observed that at higher temperature of sintering, exposure to thermal shock is damaging in general. However, the damage due to exposure to a sub-ambient temperature of -80 ºC is more damaging than that due to exposure to +80 ºC. For relatively low temperature of 540 ºC and 520 ºC, the load at rupture actually increases when exposed to +80ºC compared to the samples without any thermal shock. However, in these cases also an exposure to a sub-ambient temperature is seen to be quite damaging. Yielding load at rupture values much below the same for samples without any thermal shock. This may be due to the fact that at these temperatures (520 ºC, 540 ºC), the process of sintering might not have been completed and therefore, exposure to an elevated temperature aided in enhancement of the strength properties.

The inter-atomic diffusion between the matrix and the reinforcement as explained earlier is dependent upon temperature and time at temperature. This diffusion is responsible for the strength of the metal matrix composite and is more pronounced at elevated temperatures.[21][36] This explains the higher values of load at rupture at relatively higher temperature. This also throws light on the enhancement of strength properties, when samples sintered at relatively low temperature are exposed to an elevated temperature(+80 ºC).

It must be understood that with increase of time at sintering temperature, the time of contact of the two phases, namely the matrix and the reinforcement increases resulting in higher extents of inter-atomic diffusion which is responsible for a rigid interface. We know that there is substantial difference between the Aluminium alloy matrix and the SiC reinforcement and this mismatch gives rise to residual stress generation[37]. This relaxation of the residual stresses is limited by the extent

of inter-atomic diffusion, lower being the possibility of relaxation at a higher extent of diffusion.

With increase in both the sintering temperature and time at sintering temperature, the extent of inter-atomic diffusion increases limiting the residual stress relaxation. As a result, the residual stress concentration increases and load at rupture values are lowered when the specimen is exposed to thermal shock. This explains the load at rupture values which increases with the increase in the sintering temperature, coupled with the decrease in the holding time at the sintering temperature. A reversed trend is obtained (the load at rupture value is decreased) when the sintering temperature and the time at temperature, both are increased due to higher extents of inter-atomic diffusion and lesser possibilities of relaxation of residual stresses.

This behavior of the samples sintered at relatively lower temperature (500 ºC), away from the melting point, when exposed to thermal shocks due to exposure to +80ºC and -80ºC can be explained as follows. At lower temperatures, due to lesser extent of inter-atomic bonding, the interface is relatively weak. The strength of the interface increases as the holding time at the sintering temperature increases due to greater extent of inter-atomic diffusion. Now, when the thermal shock is due to treatment at +80ºC, there is enhancement of the interfacial bonding due to the fact that both the matrix and the reinforcement expand at +80ºC with the matrix expanding at a higher rate than the SiCp reinforcement. This enhancement is comparatively more pronounced for samples held at the sintering temperature for longer periods. Thus, at a sintering temperature of 500 º C and thermal shock due to exposure to +80ºC, the samples exhibit a higher resistance to rupture when held at the sintering temperature for longer periods. This is a physico-mechanical process.

At the same low temperature (500 ºC), the samples exposed to thermal shock at -80 ºC exhibits a decreased resistance to rupture with increase in holding time. This is because, here a reversed physico-mechanical process takes place, due to differential contraction between the matrix and the reinforcement. The one with relatively stronger interface with greater extent of inter-atomic diffusion (with longer holding time), develops higher extents of thermally degraded area at or near

the interface [38]. This whole phenomenon is due to differential contraction as a result of limited relaxation in the thermally generated residual stress.

Fig no. 18 through 20 represent the same fact through bar diagram showing the load at rupture as a function of the holding time without thermal shock as well as with thermal shocks at +80 ºC and -80 ºC respectively.

From the above, we can deduce the following:

1. At relatively higher temperatures thermal shock is more damaging for samples held at sintering temperature for longer periods.
2. The reverse happens when the time of sintering at sintering temperature is certain at relatively higher temperature of sintering.
3. The thermal shock at sub-ambient temperature is more damaging than at elevated temperature.
4. At relatively lower temperature, the resistance to rupture increases with time of holding, when the thermal shock is due to an exposure to a temperature above the ambient temperature.
5. The reverse takes place: i.e. the resistance to rupture increases with the decrese of the holding time at this relatively lower sintering temperature (500 ºC), when the thermal shock is due to exposure to sub-ambient temperature (-80 ºC).

The reason for the above critical findings can be attributed to the following in general.

a. Atomic migration or inter-atomic diffusion;
b. high residual stress due to limited or no release of the thermal residual stresses, when the interfacial bond is enhanced presenting a stronger interface;
c. Physico-mechanical processes at weaker interfaces due to misfit strain. This may be either

due to compressive residual stresses as a result of differential expansion at an elevated temperature or tensile residual stresses as a result of differential contraction at a sub-ambient temperature between the alloy matrix and the SiCp reinforcement.

4.2.2 Stress at rupture under thermal shock:

Sl no.	Temperature (ºC)	Time (hours)	Stress at rupture with different thermal exposure		
			No exposure	+80 ºC	-80 ºC
1	500	1	136.4	153.97	153.63
		2	139.8	157.4	151.1
		3	148.98	171.2	150.75
2	520	1	151	170.7	145.4
		2	163.63	169.5	148.1
		3	175.9	161	157.52
3	540	1	176.84	181.6	160
		2	177.29	193.34	171.92
		3	180.63	205.8	114.1
4	560	1	181.15	173.27	164.25
		2	182.23	177.1	153.9
		3	188.91	161.5	164.58

Table no. 5: Table showing stress values at rupture for samples at various sintering temperatures, holding times and thermal shocks given.

Fig. no. 22, fig. no. 23, fig. no. 24 show the graphical representation of stress at rupture Vs. temperature of sintering as a function of the holding time at the sintering temperatures for samples not subjected to any thermal shock and thermal shocks due to exposure to +80 ºC and -80 ºC respectively.

Fig. no. 25 through fig. no. 28 represent the data in the form of bar-diagrams for the sake of comparison between the stress values at rupture Vs. time of holding at the specific sintering temperature without any thermal shock as well as due to thermal shock with an exposure to +80 ºC and -80 ºC respectively.

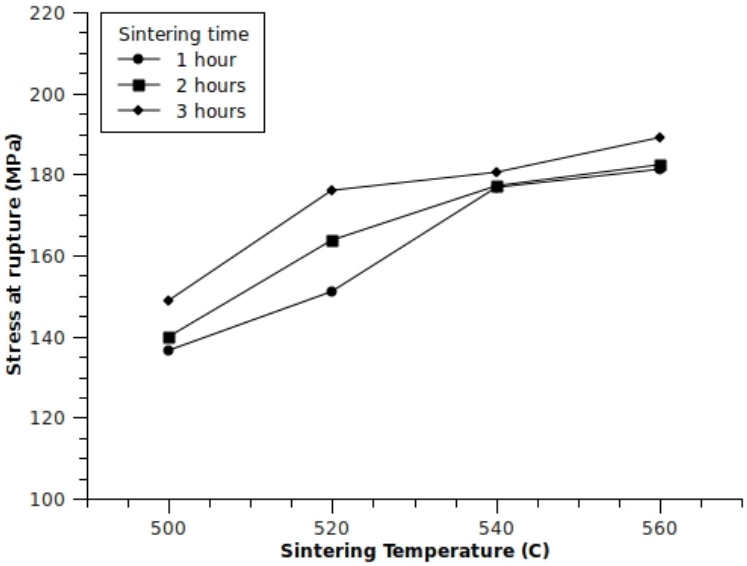


Fig no. 22:

Stress at rupture Vs. Sintering temperature as a function of Time at temperature for samples not subjected to any thermal shock.

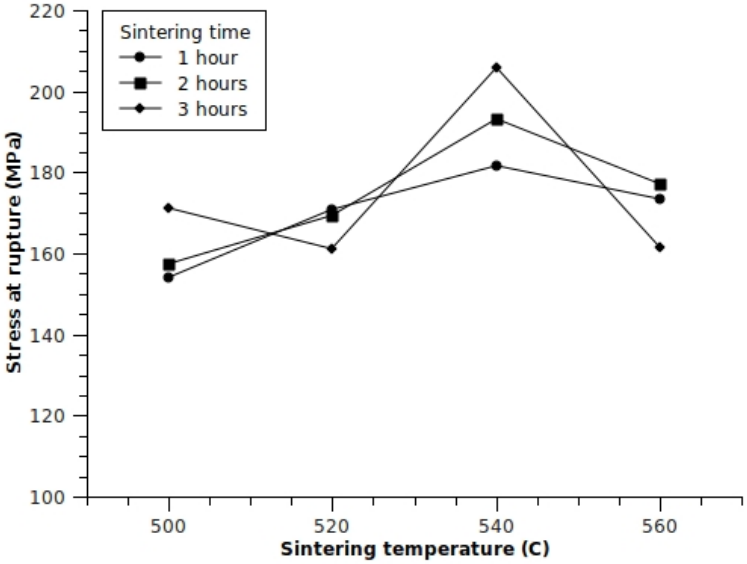


Fig no. 23:

Stress at rupture Vs. Sintering temperature as a function of Time at temperature for samples subjected to an elevated temperature of +80 ºC.

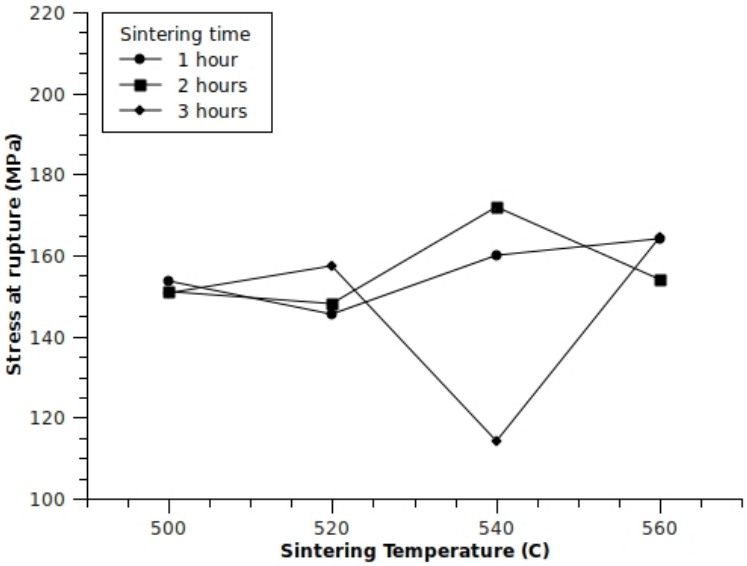


Fig no. 24:

Stress at rupture Vs. Sintering temperature as a function of Time at temperature for samples subjected to a sub-ambient temperature of -80ºC.

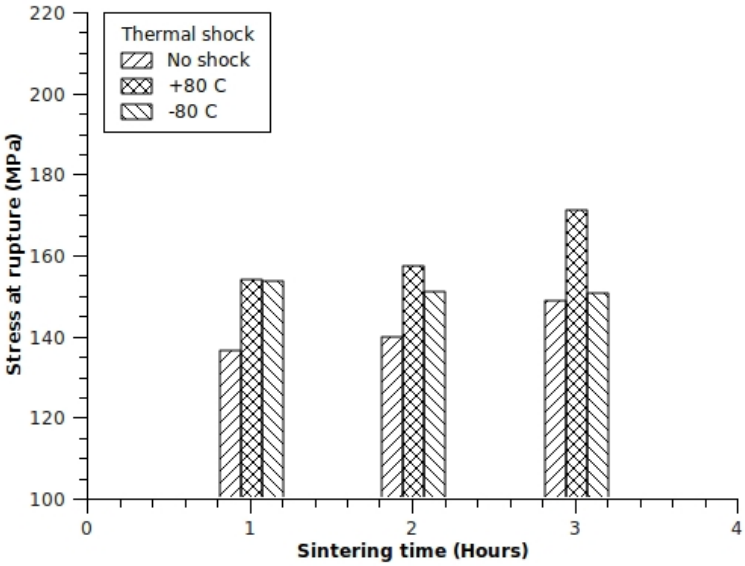


Fig no. 25:
Stress at rupture Vs. Time at sintering temperature (500 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

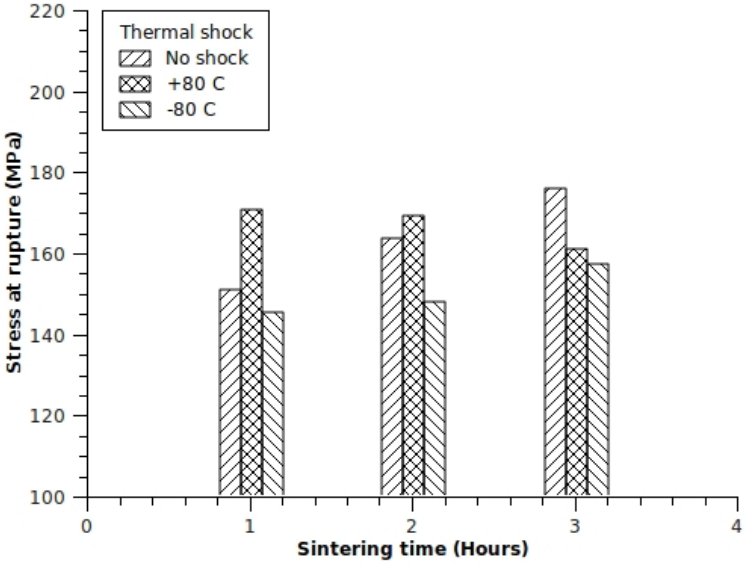


Fig no. 26:
Stress at rupture Vs. Time at sintering temperature (520 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

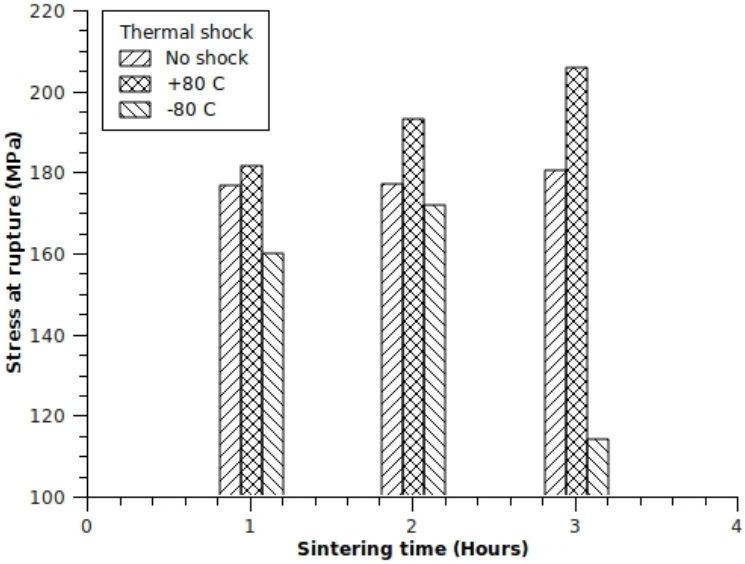


Fig no. 27:
Stress at rupture Vs. Time at sintering temperature (540 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

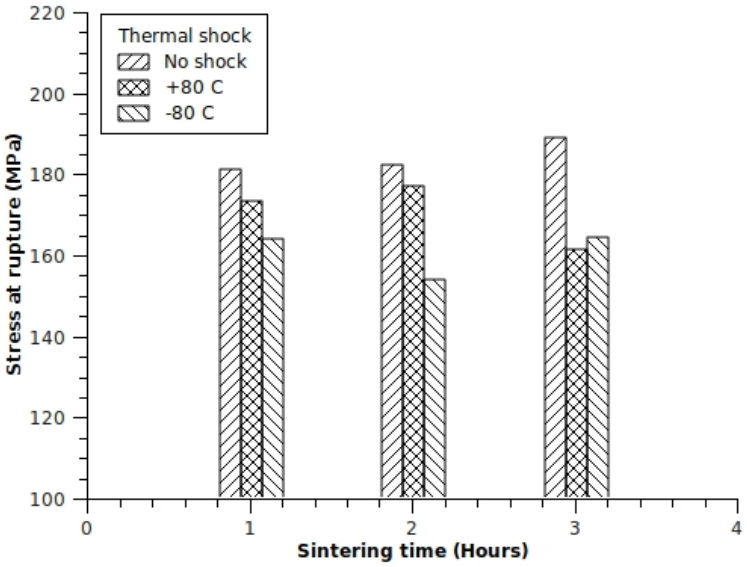


Fig no. 28:
Stress at rupture Vs. Time at sintering temperature (560 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

The above diagrams, as expected, show a variation of stress at rupture with respect to time and thermal shock similar to those in the case of load values at rupture, since stress is the load per unit area. We can, however, note the following from the experimental findings:

1. At relatively higher sintering temperatures and for shorter-term use, (i.e. when the specimen are exposed to the sintering temperature for short durations), the thermal shock is no much damaging.
2. At relatively higher sintering temperatures and long-term use, i.e. longer exposure, the thermal shock is found to be more damaging.
3. At relatively lower temperatures, close to the melting point of the matrix, the MMC responds well, while put to long-term use even when exposed to thermal shock at an elevated temperature.
4. For shot-term use, the thermal shock at an elevated temperature is more damaging for samples sintered at relatively lower temperatures.
5. For long term use, the thermal shock due to a sub-ambient temperature is more damaging when test specimen is sintered at relatively lower temperatures.

4.2.3 Displacement at rupture:

Sl no.	Sintering temperature (ºC)	Time at temperature (Hours)	Displacement at rupture under different thermal exposure		
			No shock	Exposure to +80 ºC	Exposure to -80 ºC
1	500	1	0.8129	0.7323	0.7111
		2	0.8129	0.6316	0.5383
		3	0.4174	0.5822	0.4431
2	520	1	0.7049	0.7818	0.6774
		2	0.8257	0.7287	0.6078
		3	0.7342	0.6683	0.5950
3	540	1	1.0880	0.8330	0.5090
		2	0.9026	0.7708	0.7403
		3	0.8221	0.7604	0.7554
4	560	1	0.6262	0.8550	0.8112
		2	0.7781	0.8300	0.8236
		3	1.0950	0.8253	0.8247

Table no. 6: Table showing displacement values at rupture for samples at various sintering temperatures, holding times and thermal shocks given.

Table no. 5 represents the experimental data for displacement at rupture for the samples without thermal exposure and with thermal exposure at an elevated temperature (+80ºC) and sub-ambient temperature (-80 ºC). The same data is plotted in fig. no. 29, fig. no. 30 and fig. no. 31 which show the variation of displacement at rupture values as a function of temperature of sintering for different holding times at the sintering temperature when exposed to thermal shock as well as without any exposure to thermal shock.

Fig. no. 32 through 35 present the displacement at rupture values versus the time at sintering temperature for various groups of samples which are not exposed to any thermal as well as those exposed to elevated (+80 ºC) and sub-ambient (-80 ºC) temperatures.

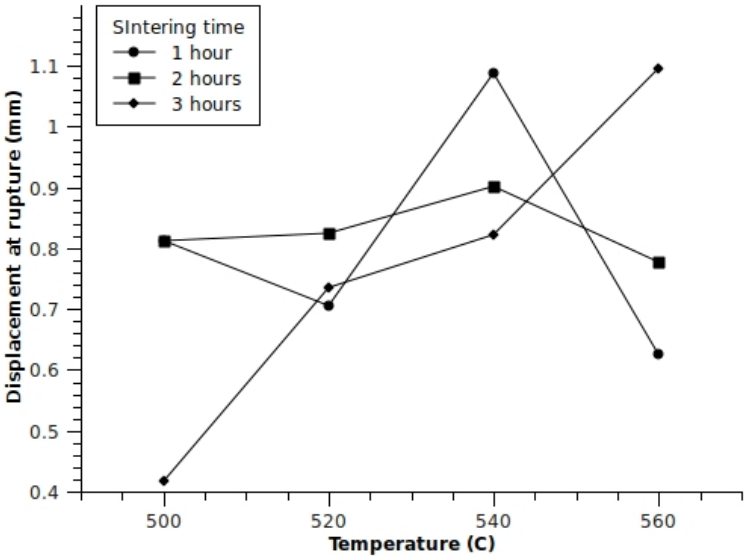


Fig no. 29:
Displacement at rupture Vs. Sintering temperature as a function of Time at temperature for samples not subjected to any thermal shock.

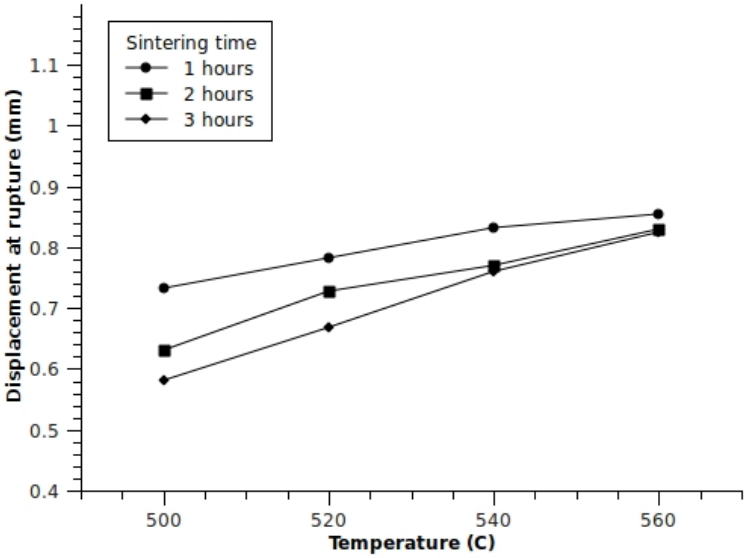


Fig no. 30:
Displacement at rupture Vs. Sintering temperature as a function of Time at temperature for samples subjected to thermal shock at elevated temperature of +80 ºC.

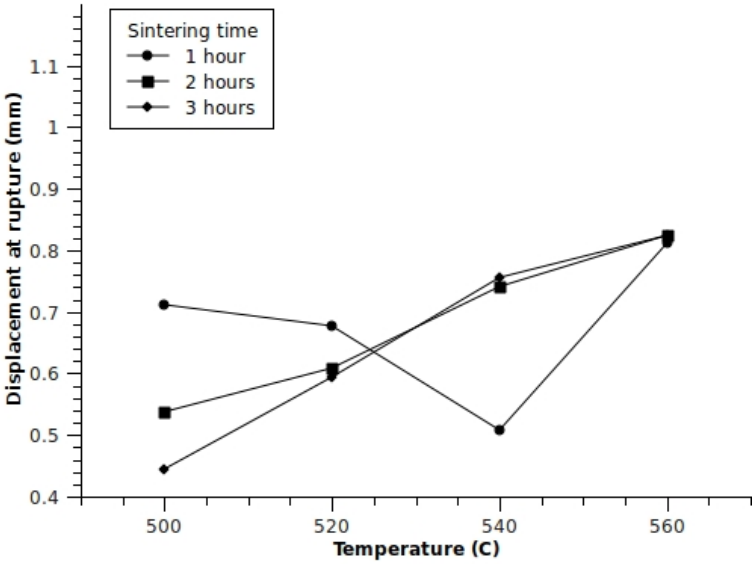


Fig no. 31:
Displacement at rupture Vs. Sintering temperature as a function of Time at temperature for samples subjected to thermal shock at sub-ambient temperature of -80 ºC

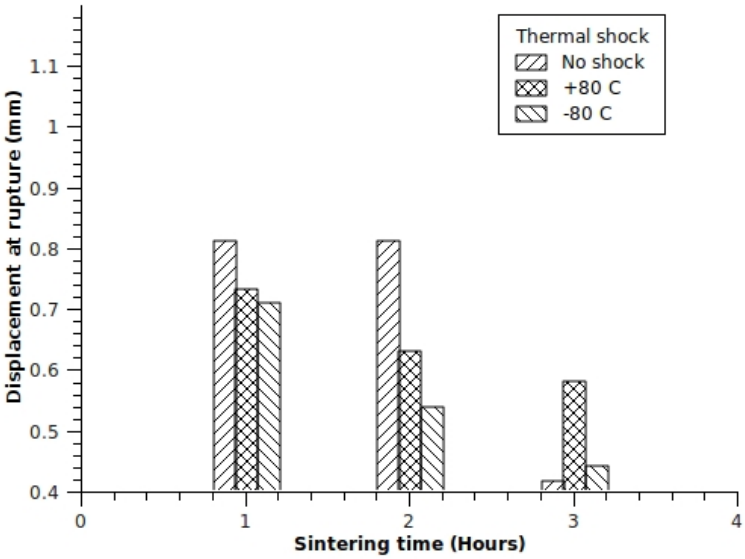


Fig no. 32:
Displacement at rupture Vs. Time at sintering temperature (500 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

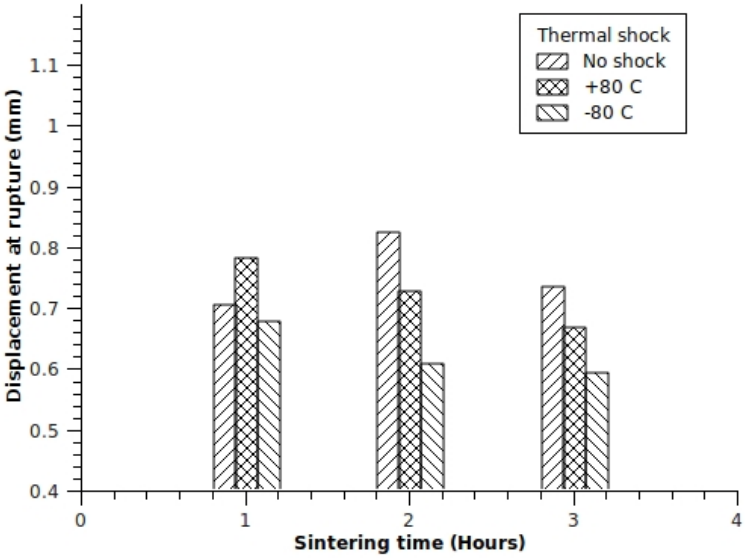


Fig no. 33:
Displacement at rupture Vs. Time at sintering temperature (520 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

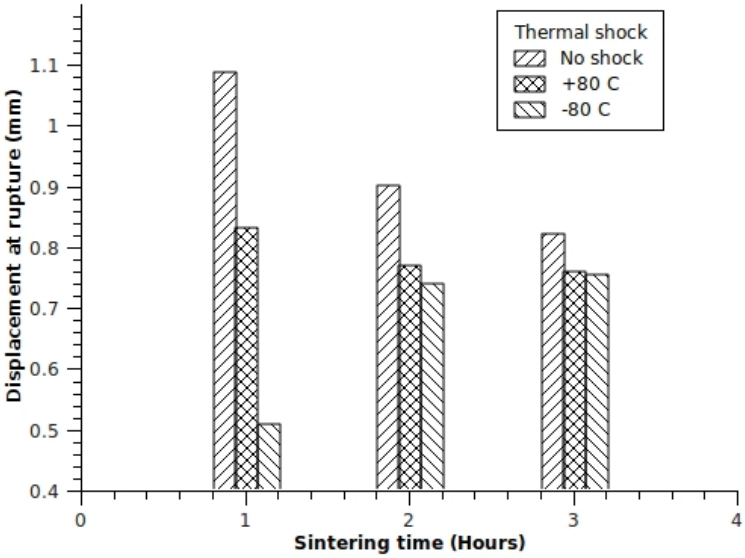


Fig no. 34:
Displacement at rupture Vs. Time at sintering temperature (540 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

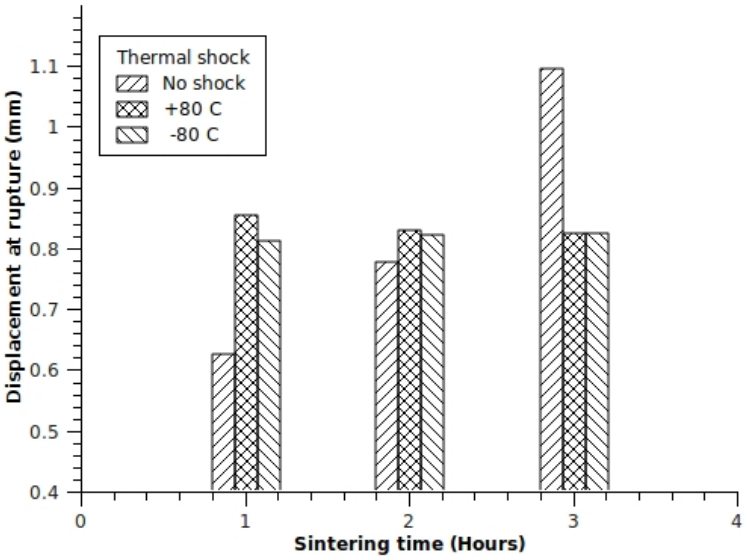


Fig no. 35:
Displacement at rupture Vs. Time at sintering temperature (560 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

The displacement values represent the total deformation undergone by the specimen before it finally gives up to the load and rupture is initiated. It must be understood at the very out set that the displacement/deformation depicts the plastic deformation of the specimen at crack initiation. A ductile specimen free from stresses undergoes a larger deformation before crack initiation. On the other hand, a stressed specimen ruptures, i.e. surrenders to the load with little or no displacement/plastic deformation. The stress in the specimen is mainly due to the residual stresses as a result of the difference in the CTE of the matrix and the SiC particulates. This mismatch may be due to the thermal shock at an elevated temperature of +80 ºC or a sub-ambient temperature of -80 ºC. Even, as suggested by Liu, et al. [37], in these particulate reinforced MMCs the mismatch in the CTE gives rise to residual stresses in the matrix during fabrication of the composite itself, without the specimen being subjected to any thermal treatment.

The samples sintered at relatively high temperature (560 ºC) show and increase in the total displacement values with the increase in the holding time at sintering temperature. We know that irrespective of the sintering temperature, the inter-atomic diffusion gets enhanced with the time of holding. This generates a strong interface which acts as a constraining factor for matrix deformation, i.e. the displacement at high temperature with the increase of holding time should have decreased. However, as presented earlier in our case, the trend of variation is reversed. Mummery,

et al. [39] suggest that in the case of a strong deformation for specimen with strong interfacial bonding (as in this case), particle cracking can be considered as the only factor responsible for the increase in the ductility. In this case, the increase in the ductility may be due to particle cracking, which can be the only way of stress release. In the case of very low temperature, away from the melting point, for samples without any thermal shock, the experimental findings show a slight lowering in the plastic deformation at longer time of holding at sintering temperature (3hours) compared to shorter times of holding (1 and 2 hours). It is expected that at this lower temperature (500 ºC), the interfacial bonding is not very strong. However, a decreased value of the displacement for the sample held for 3 hours at sintering temperature might have resulted in a relatively strong interface which restrains plastic deformation.

This trend, i.e. lowering of displacement with increase in the time of holding is clearly seen for samples sintered at a relatively higher temperature of 540 ºC. This can be explained through the formation of a strongly bonded interface at this temperature with the increase in holding time which is responsible for the limited deformation with increase in holding time. On the other hand, a slightly low temperature of 520 ºC which is not as low as 500 ºC, does not show any clear trend of variation of dislocation values with increase in holding time. This may be due to the fact that at this temperature, the bond at the interface is not very well defined.

The data in table no. 5 and the related graphs and bar diagrams concerned with thermal shock due to exposure to an elevated temperature of +80 ºC as well as a sub-ambient temperature of -80 ºC clearly show the following trends.

a. Irrespective of the sintering temperature, the total deformation undergone by the test piece is lowered with the increase of the holding time.
b. At relatively high temperature of sintering, there is an increase in extent of deformation with the increase of the holding time with an exposure of the sample to a sub-ambient temperature (-80 ºC).

c. For relatively low temperature, the displacement is seen to decrease with increase in holding time for samples exposed to a sub-ambient temperature of -80 ºC.
d. In all cases of temperature and time of holding, the deformation/displacement assumes a lower value with an exposure to a sub-ambient temperature (-80 ºC) compared to that at elevated temperature (+80 ºC).

These trends in the displacement/deformation values can be explained in the basis of the residual stresses generated due to CTE mismatch between the matrix and the reinforcement as well as the release of the stress either due to the plastic deformation of the matrix or the cracking of the particulate reinforcements.

As explained earlier [37], the mismatch in the CTE between the matrix and the reinforcement gives rise to residual stresses in the matrix during fabrication of the composite. In addition to this, when the composite is exposed to an elevated temperature (+80ºC in this case), the development of residual stresses gets enhanced. Therefore, irrespective of the sintering temperature, the inter-atomic diffusion assumes a greater value with the increase in the time of holding at the sintering temperature, consequently generating a strong interface. This strong interface acts as a constraining factor for matrix deformation [39. Therefore, high residual stresses are generated. This is because the redistribution of these stresses is limited due to the increases interfacial strength and the constrained plastic flow of the alloy matrix [2]. This explains the decrease in the total deformation/displacement of the specimen before rupture sets in, with the increase in the holding time at the sintering temperature irrespective of the sintering temperature examined.

With an exposure at a sub-ambient temperature, the total deformation values prior to rupture are increased when the sintering temperature is relatively high (560ºC) and the holding time at the sintering temperature is increased for this high temperature.

At a sub-ambient temperature, the Aluminium alloy matrix contracts to a greater extent

compared to the SiC particulates. This mismatch in the CTE leads to the generation of residual stresses. The only possibility that these cryogenic stresses can be relieved is through the cracking of the particulate reinforcement or because of the presence of the discontinuities. However, the redistribution of the cryogenic residual stresses is limited due to the high temperature of sintering and the longer time of holding at the sintering temperature. Therefore, the only possibility of stress relief is reinforcement cracking, and the increase in the total deformation in this particular case may be explained by the cracking of the particulate reinforcement. This phenomenon is supported by the claim put forth by L. Llorea, et al. [9]. They suggest that in the initial stages if plastic deformation, the increase in the load carried by the particulate reinforcement is mainly due to the progressive strain hardening of the ductile material. As the matrix strain hardening capacity is saturated, due to increase in the cryogenic residual stress in this case, relaxation of stresses result from the fracture of the particulate reinforcement. This particulate reinforcement fracture is responsible for the stress transfer to the nearby particles causing greater particle fracture which brings in the final failure of the composite.

At relatively low temperature of sintering, however, the total deformation undergone by the composite prior to rupture decreases with the increase in holding time at the sintering temperature when the specimen are exposed to a sub-ambient temperature (-80 ºC in this case). This phenomenon can be attributed to the generation of residual stress due o the mismatch in the CTE between the matrix Al-alloy and the SiCp reinforcement. This residual stress can not get redistributed due to the presence of a relatively strong interface which is developed with the increase in the holding time at sintering temperature. Thus, the plastic deformation of the matrix is constrained and the total percentage of deformation is limited.

4.2.4 Strain values at rupture :

Sl no.	Sintering temp. (ºC)	Time at temp. (Hours)	Strain at rupture under different thermal exposure (%)		
			No thermal shock	+80 ºC	-80 ºC
1	500	1	3.226	2.929	2.802
		2	3.183	2.501	2.086
		3	1.624	2.318	1.816
2	520	1	2.766	3.063	2.646
		2	3.277	2.869	2.389
		3	3.143	2.633	2.321
3	540	1	4.278	3.280	2.935
		2	3.690	3.152	2.942
		3	3.275	3.093	2.985
4	560	1	2.419	3.379	3.253
		2	3.163	3.320	3.320
		3	4.397	3.262	3.319

Table no. 7: Table showing strain values at rupture for samples at various sintering temperatures, holding times and thermal shocks given.

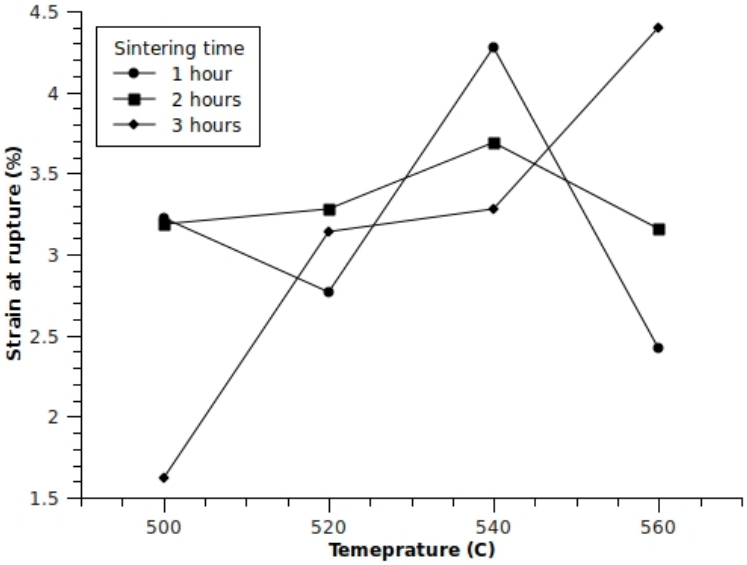


Fig no. 36:
Strain at rupture Vs. Sintering temperature as a function of Time at temperature for samples not subjected to any thermal shock.

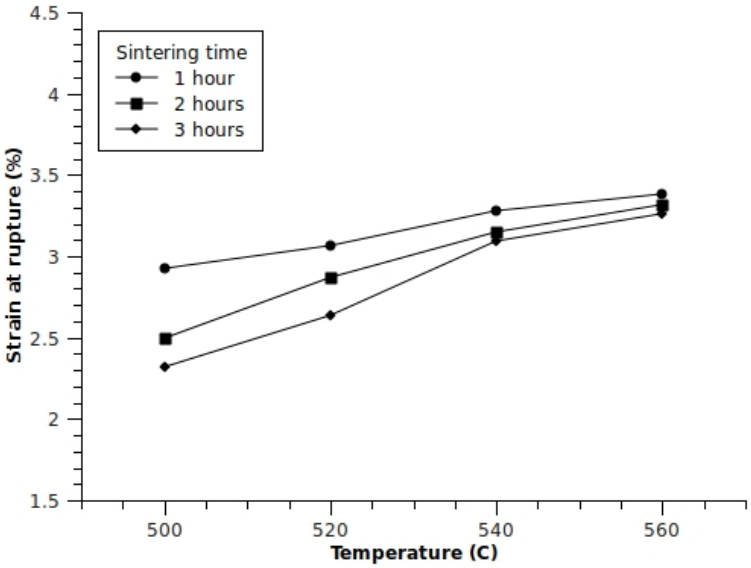


Fig no. 37:
Strain at rupture Vs. Sintering temperature as a function of Time at temperature for samples subjected to thermal shock at elevated temperature of +80 ºC

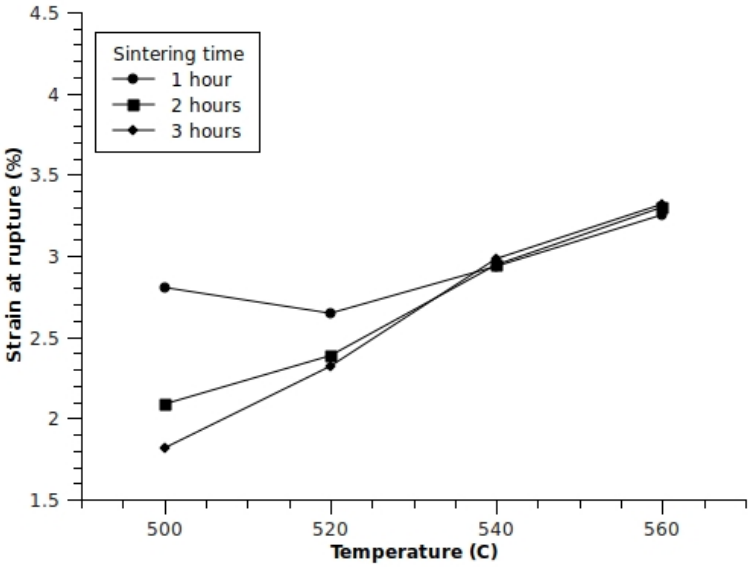


Fig no. 38:
Strain at rupture Vs. Sintering temperature as a function of Time at temperature for samples subjected to thermal shock at sub-ambient temperature of -80 ºC

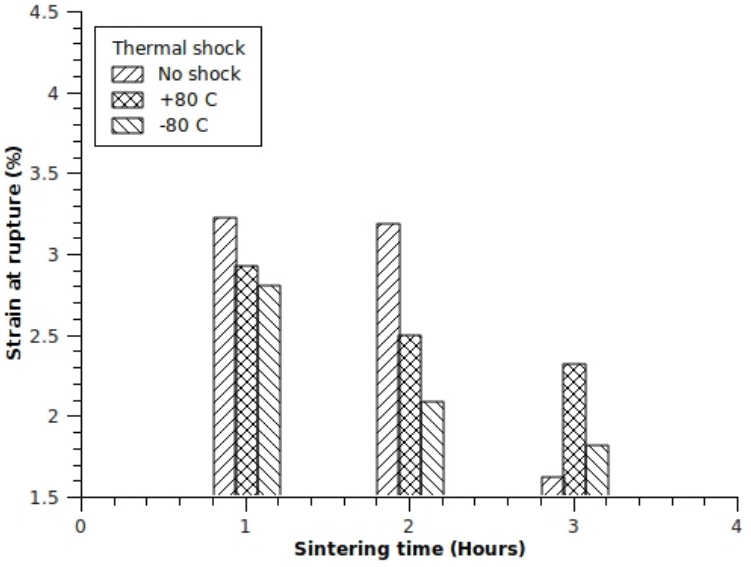


Fig no. 39:
Strain at rupture Vs. Time at sintering temperature (500 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

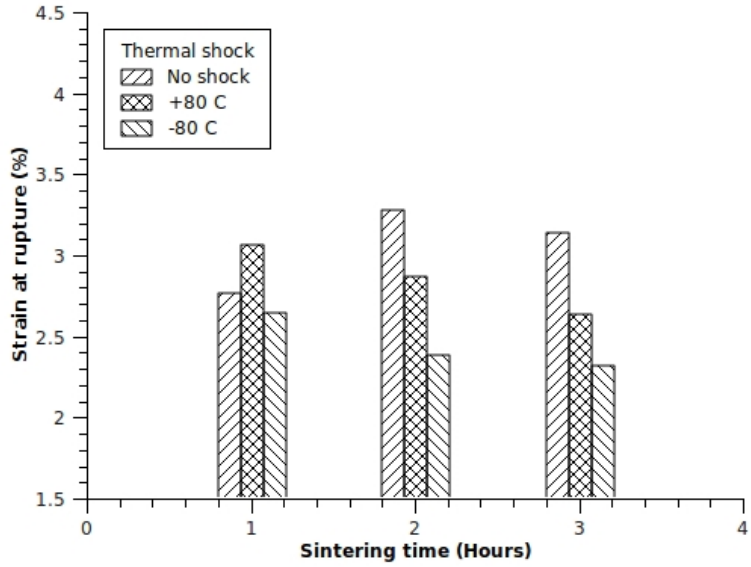


Fig no. 40:
Strain at rupture Vs. Time at sintering temperature (520 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

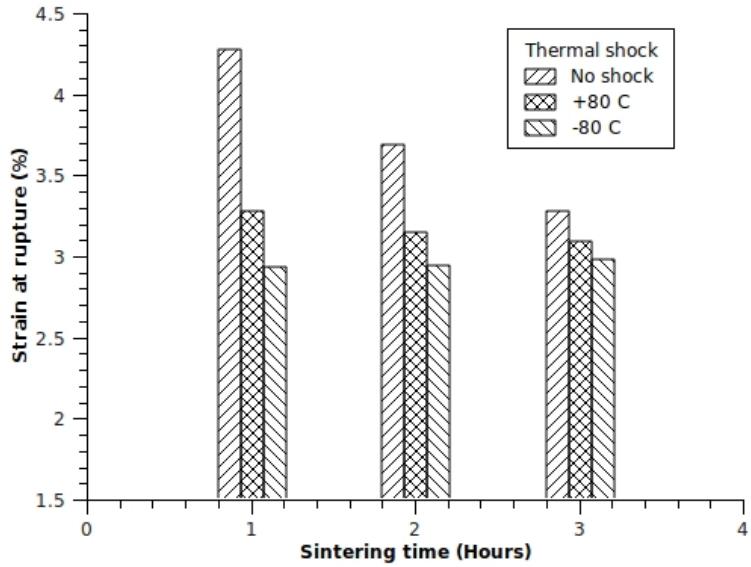


Fig no. 41:
Strain at rupture Vs. Time at sintering temperature (540 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

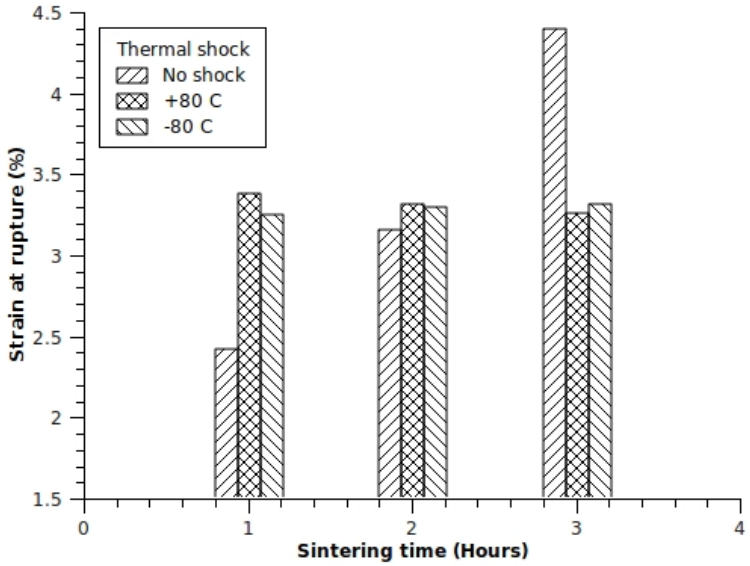


Fig no. 42:
Strain at rupture Vs. Time at sintering temperature (560 ºC) for samples not subjected to thermal shock and subjected to thermal shocks at +80 ºC and -80 ºC.

Table no. 6 houses the experimental findings for the strain at rupture as a percentage of the original length for test specimen without any thermal exposure and with thermal exposure to +80 ºC and -80 ºC respectively.

The same data is presented in a practical form in fig. no. 36, fig. no.37 and fig. no. 38 which present the data plotted Vs the temperature as a function of time. Fig. no. 39 through fig. no. 42 present the same data in the form of bar-diagrams so that a direct comparison can be made for the variation of the data under different conditions of experimentation.

A material with a higher value of strain has a higher ductility and has a greater ability to deform before failing under load. Strain being the change in length per unit length of the material exposed to the load, it must be appreciated that the percentage strain as recorded in table no. 6 and plotted in the diagrams mentioned above, has a direct link with the total displacement undergone by the material before rupture.

As can be seen from the data, the percentage strain variation exhibit a similar trend as those exhibited by the total deformation/displacement data. This trend can be attributed to similar factors as discussed earlier in the case of displacement variations.

Ductility, as can be appreciated, is a measure of the ability of the material to undergo deformation before fracture sets in. The conventional measure of ductility is the engineering strain at fracture or the change in length divided by the original length or simply the percentage strain. This fact answers the question why the percentage strain in rupture is an important factor concerning material property which must be considered while designing an engineering component. A material characterized by lower values of percentage strain at fracture will generate an engineering component which will catastrophically give away under load without giving any warning. On the other hand a material characterized by higher values of percentage strain will get deformed as a consequence of the applied load, give a warning and then fail. In light of the above, toughness of the material may provide an important parameter for designing purposes. The key to

toughness is a good combination of strength and ductility. A material with high strength and high ductility will have higher toughness values compared to one with low strength and high ductility.

On the basis of the above, we can note the following from the results obtained int his project work. We can consider both, the stress at rupture or simply the capacity of the material to absorb energy before fracture and the percentage strain or ductility or simply the ability of the material to deform plastically before fracture. We can infer the following:

(i) Exposure to an elevated temperature, i.e. a temperature above the ambient, is not much damaging for short term use of the material when the sintering temperature is relatively high.

(ii) Exposure to a sub-ambient temperature is not much damaging for short-term use of the material when the sintering temperature is relatively low.

(iii) When long term use is concerned, either of the two, i.e. the ability to absorb energy and the ability to deform plastically before rupture, or both of them do not exhibit good results when the material is exposed to thermal shock at an elevated temperature or even sub-ambient temperature. Thus the toughness values are lowered restricting the use of the material for fabrication of load bearing engineering components.

(iv) An exposure to a sub-ambient temperature for all cases of sintering temperature and time of holding at the sintering temperature, yields a lower value of the total displacement at rupture compared to an exposure to an elevated temperature (above the ambient). This may be attributed to the difference in the CTE of the alloy matrix and the SiCp reinforcement which cause the generation of residual stresses. These residual stresses restrict the plastic flow of the matrix thereby lowering the total displacement values before fracture sets in.

4.3 Assessment and evaluation based on SEM micrograph study:

The thermal shock induced residual stresses caused due to a mismatch in the CTEs of the alloy matrix and the ceramic reinforcement control the mode of failure in the MMC. It must be understood that the interface in MMC is a porous non-crystalline portion in the composite when compared to either the metal matrix or the ceramic reinforcement. In the present case the thermal shock, both due to exposure to an elevated temperature or to a sub-ambient temperature develop the residual stresses and these induced stresses are tensile stresses in alloy matrix and compressive stresses in the SiCp reinforcement, due to the CTE mismatch.

Extensive micrographs generated using SEM(JSM-6480 LV) have been studied to explain the failure mode which is prevalently controlled by the extent and the type of residual stresses developed. These residual stresses dictate the mode of failure which influence the interfacial bonding, decohesion, etc. and thus influence the mode of failure.

The state of residual stresses is mostly dynamic and a relaxation of these stresses is caused due to one or many of the following phenomena.

1. Interface sliding
2. Decohesion
3. plastic strain
4. Micro and Macro-cracking
5. Particulate breaking/cracking.

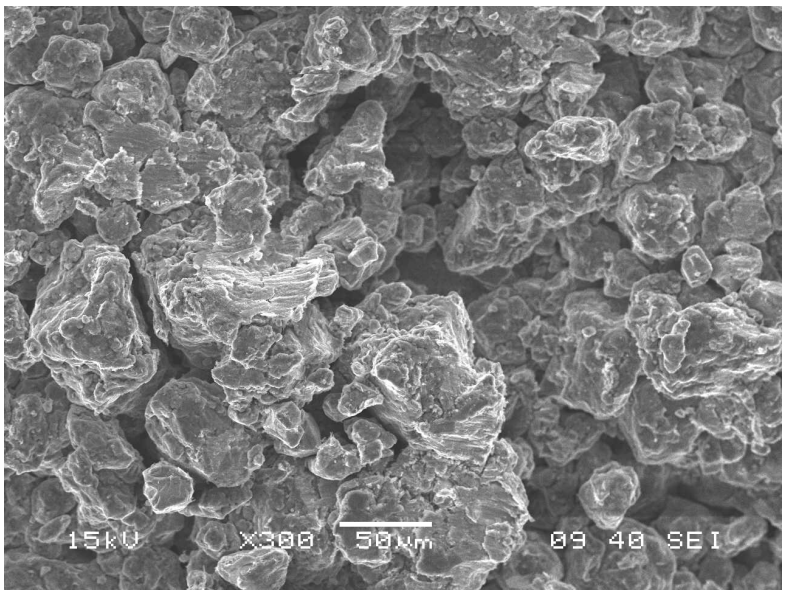
Stresses induced due to the difference in the CTE of the matrix and the reinforcement may impart plastic deformation to the matrix which results in a reduction in residual stresses. In a similar manner the thermal mismatch strain often develops cracks in the matrix, again resulting in a relaxation of the residual stresses. Witners et al. claim that residual stresses may be relaxed, i.e. the

relaxation of the residual stresses may be caused when the time gap is long, i.e. for long term use even at room temperature. On the other hand reversal between high or low temperature including sub-ambient temperature and the room temperature can also modify the state of the residual stresses.

The interface in the MMC being porous and non-crystalline, residual stresses are readily relaxed here. Therefore, the following two generalized observations are interesting for record.

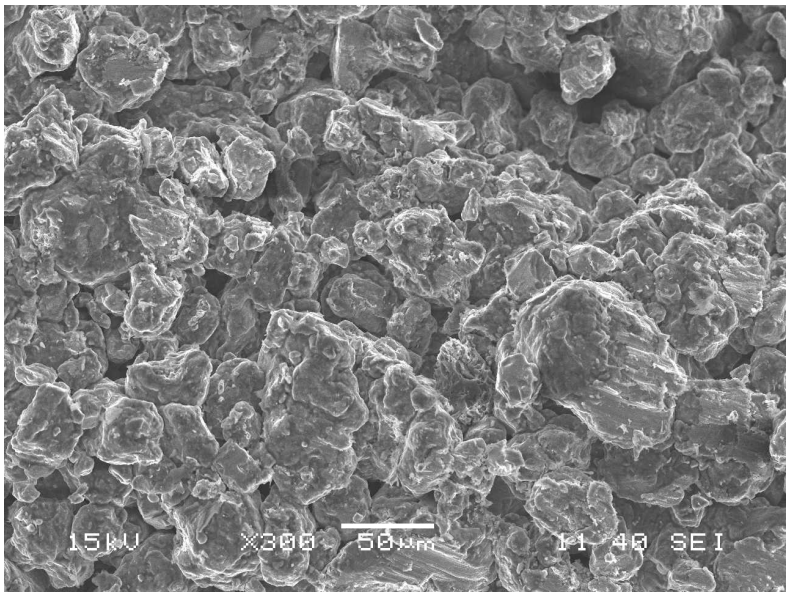
i. When the particle density is high, meaning, interface availability is plenty, failure is due to the formation and propagation of cracks at the interfacial region. This type of failure proceeds by the nucleation of voids and subsequent coalescence of these voids of the cracks leading to failure.
ii. When the particle density is low, i.e. in regions which are particle starved, meaning that the interface is limitedly available, particle failure is predominating resulting in a final failure of the composite.

The representative micrographs are presented through SEM analysis in micrograph no.1 through micrograph no. 12. Micrograph 1 and 2 clearly show the voids which would subsequently be coalesced resulting in the formation and propagation of crack leading to failure.



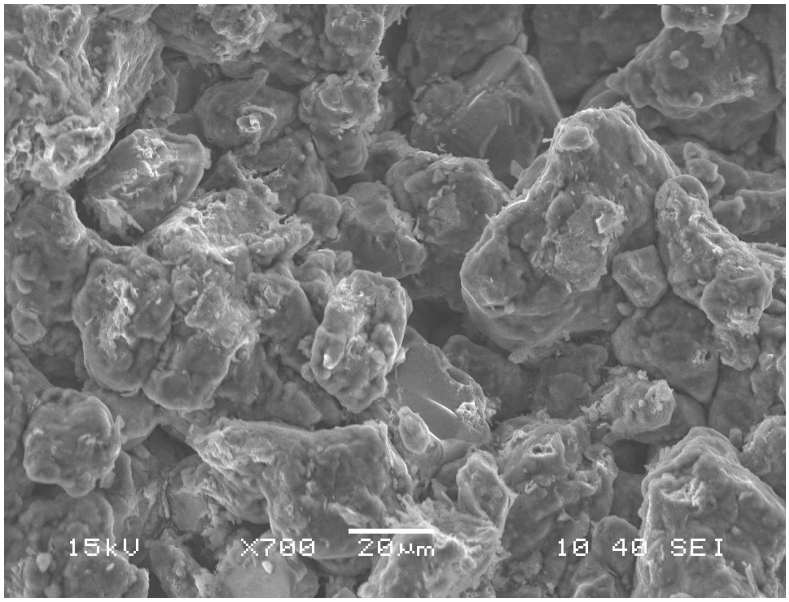
Micrograph no. 1

Temp: 560 ºC
Time: 1 hour
Treatment: None



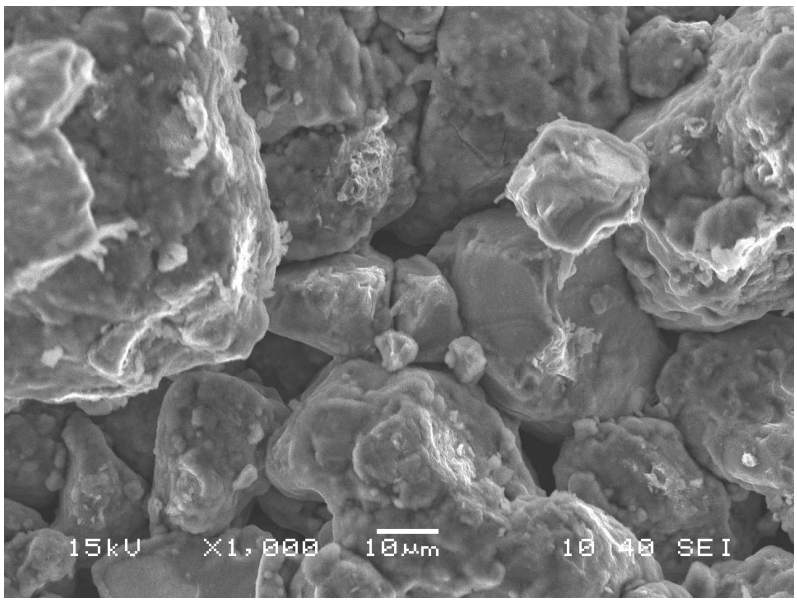
Micrograph no. 2

Temp: 560 ºC
Time: 3 hours
Treatment: None



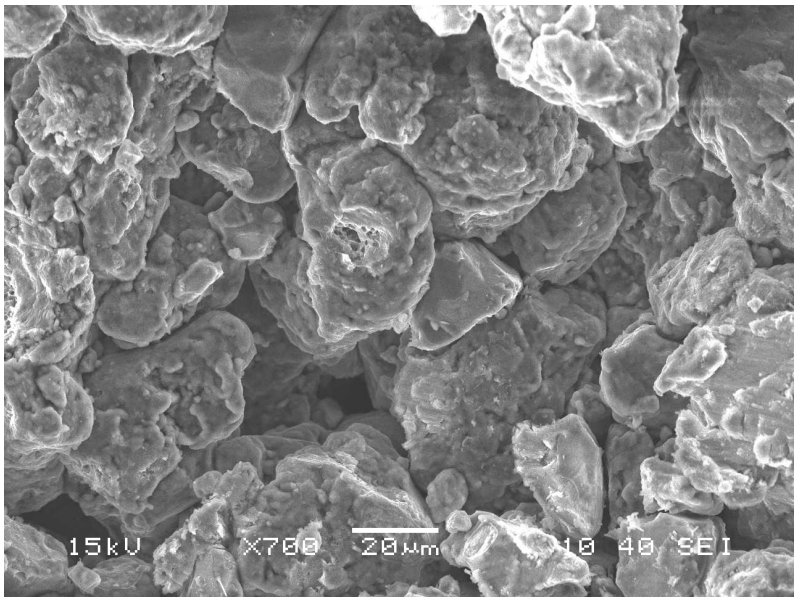
Micrograph no. 3

Temp: 560 ºC
Time: 2 hours
Treatment: - 80 ºC



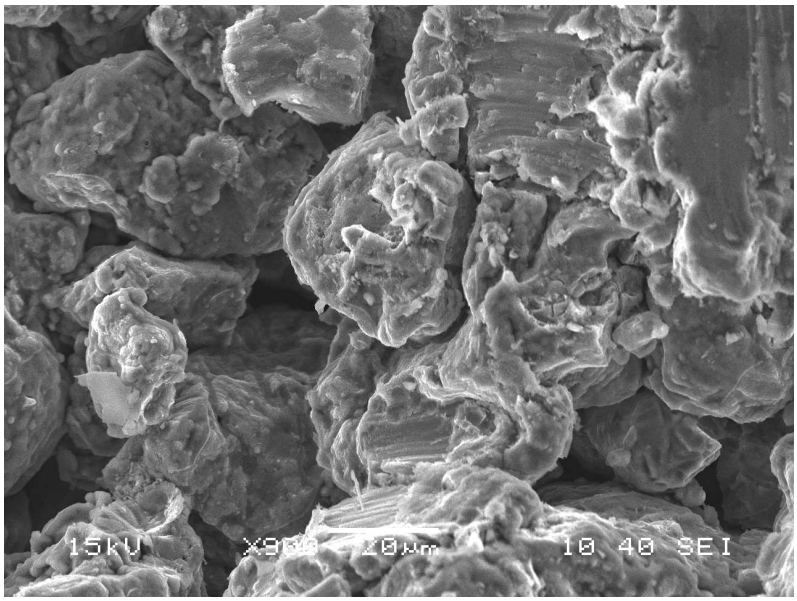
Micrograph no. 4

Temp: 560 ºC
Time: 2 hours
Treatment: - 80ºC



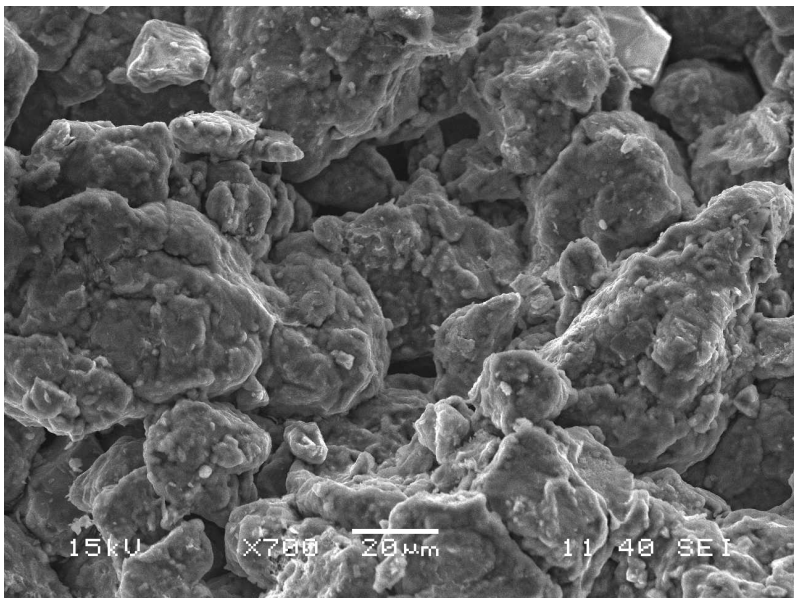
Micrograph no. 5

Temp: 540 ºC
Time: 1 hour
Treatment: - 80ºC



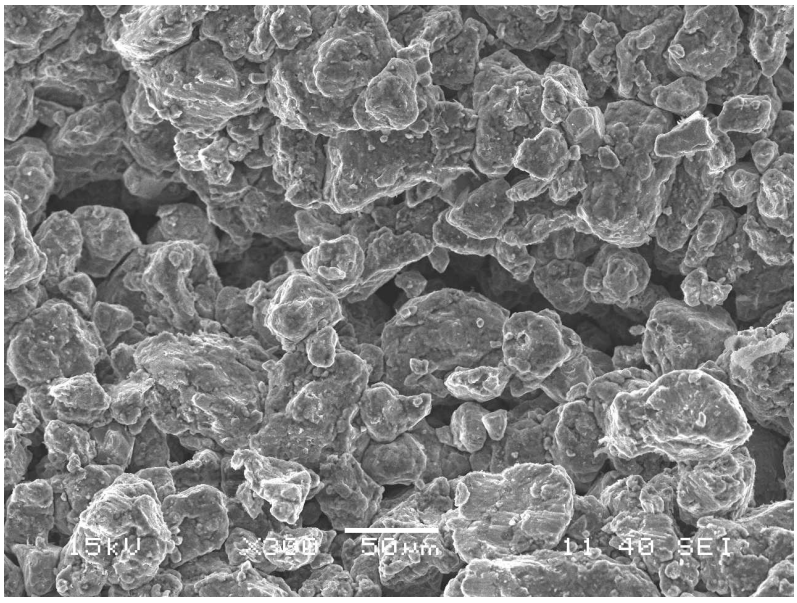
Micrograph no. 6

Temp: 540 ºC
Time: 1 hour
Treatment: - 80ºC



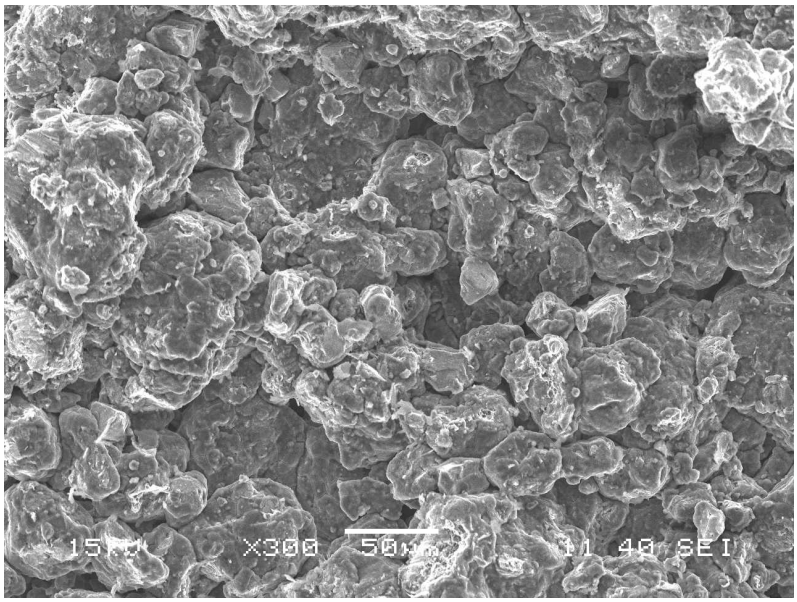
Micrograph no. 7

Temp: 520 ºC
Time: 3 hours
Treatment: - 80 ºC



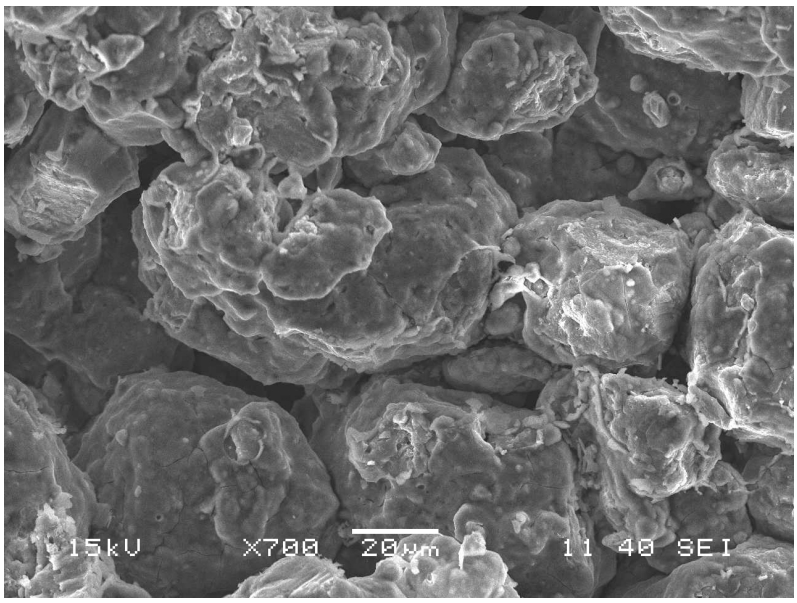
Micrograph no. 8

Temp: 520 ºC
Time: 2 hours
Treatment: + 80ºC



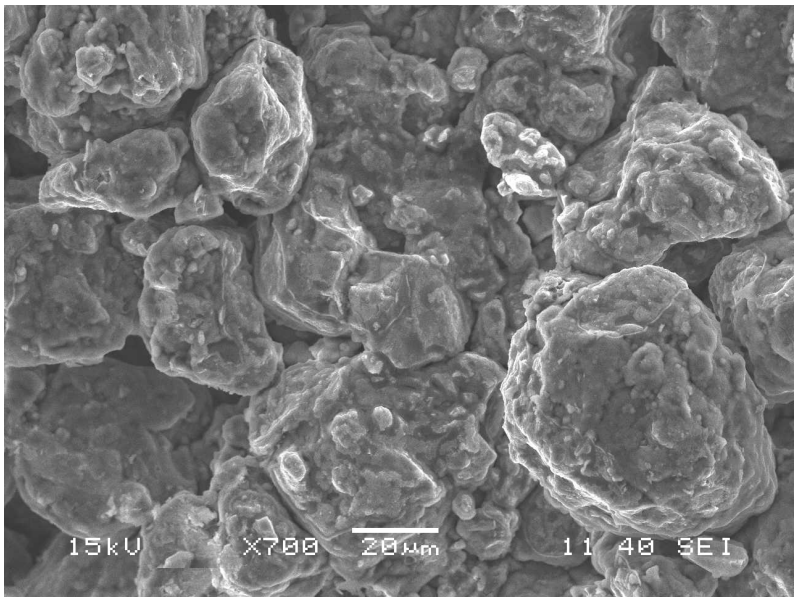
Micrograph no. 9

Temp: 540 ºC
Time: 2 hour
Treatment: +80ºC



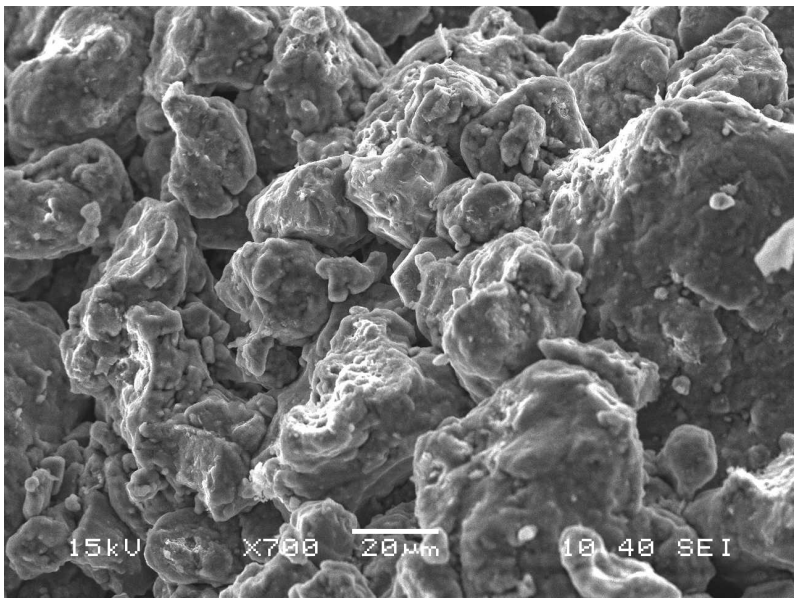
Micrograph no. 10

Temp: 540 ºC
Time: 3 hours
Treatment: - 80ºC



Micrograph no. 11

Temp: 500 ºC
Time: 1 hour
Treatment: - 80ºC



Micrograph no. 12

Temp: 560 ºC
Time: 3 hours
Treatment: - 80ºC

Micrograph no. 3 and 4 reveal matrix as well as particle failure. The exposure to -80 ºC makes the ductile alloy matrix brittle. This might have resulted from a contraction of the matrix due to exposure to the sub-ambient temperature.

Micrograph no. 6 shows the effect of the outcrop due to contraction of the matrix as a result of an exposure to the sub-ambient temperature (-80ºC). The outcrop leads to coalescence of micro-voids effectively and gives way to the generation of potential micro-voids. This phenomenon, i.e. the generation of potential micro-voids leads to a reduced threshold energy giving way to the cracks to be generated for their subsequent propagation.

An analysis of micrograph no. 7 reveal that the points as noted against micrograph no. 6 leading to the generation of cracks are less global. These revelations, i.e. the outcrop leading to the crumbling of the matrix, not being global, is more damaging to the microstructure. Micrograph no.7 also reveals some cracks developed in the particulate reinforcements.

Micrograph no. 8 and 9 reveal matrix failure. Here incomplete sintering, either due to a relatively low temperature of sintering or a lower time of sintering at the sintering temperature has resulted in improper anchor of the particulates. Therefore, strengthening mechanism has not been much effective and exposure to the elevated temperature (+80ºC) leads to matrix failure. In this case, stress is not being transferred to the particles due to the weak bonding between the matrix and the reinforcement.

Micrograph no. 10 shows interfacial failures due to the sub-ambient temperature (-80 ºC). Here the interfacial stress transfer seems to be more effective.

Micrograph no. 11 and 12 show predominating particle failure. This may be due to stress transfer from matrix to the particulates due to cryogenic conditioning (-80 ºC). These micrographs clearly show that the concerned regions are particle starved regions. Thus making limited availability of the interface which encourages particle failure, stress being transferred from the

matrix to the particulate.

On the basis of the above micrograph studies, it can be safely concluded that,

i. When the thermal shock is due to exposure to elevated temperature, the dominating failure mode is cavity generation. At this high temperature, inter-diffusion is high, resulting in strong bonding. Therefore, here failure is primarily due to generation of discontinuities, i.e. cavities at the interface.

ii. When the thermal shock is due to an exposure to a sub-ambient temperature, the dominating failure mode is interfacial failure and/or matrix damage.

CHAPTER 5
CONCLUSIONS

5. CONCLUSIONS:

From the project work, the following conclusions can be arrived at, based on the experimental findings and the detailed discussions made.

A. The strength and ductility properties of MMCs under investigation depend on both, the sintering temperature and the holding time of the sintering temperature.

B. Both the strength and ductility properties get altered for all temperatures of sintering and holding time at temperature when the as-sintered specimen are exposed to thermal shock, whether the shock is due to an elevated temperature (above the ambient) or due to sub-ambient temperature (below the ambient).

C.

1. From the strength point of view, the following can be inferred.
    i. At relatively higher sintering temperatures and for short-term use, (i.e. when the specimen are exposed to the sintering temperature for short duration), the thermal shock is not much damaging.
    ii. At relatively higher sintering temperatures and long-term use (i.e. longer exposure), the thermal shock is found to be more damaging.
    iii. At relatively lower temperatures, close to the melting point of the matrix, the MMC responds well while put to long-term use, even when exposed to thermal shock at an elevated temperature.
    iv. For short-term use, the thermal shock at an elevated temperature is more damaging for samples sintered at relatively lower temperature.
    v. For long-term use, the thermal shock due to a sub-ambient temperature is more damaging when test specimen is sintered at relatively lower temperature.
2. From the ductility point of view, the following can be inferred.
    i. Irrespective of the sintering temperature, the total deformation undergone by the test piece is lowered with the increase in the holding time.

ii. In all cases of temperature and time of holding, the deformation / displacement assumes a lower value when the specimen of the MMC are exposed to sub-ambient temperature compared to that at elevated temperature.

D. The generation of residual stresses as a result of thermal mismatch are responsible for the failure of the MMCs.

E. It can further be inferred that:

i. For short-term use of the MMCs sintered at a relatively high temperature, the thermal shock due to exposure to an elevated temperature is less damaging.

ii. For short-term use of the MMCs sintered at lower temperatures away from the melting point of the alloy matrix, thermal shocks are less damaging when caused due to exposure to a sub-ambient temperature.

iii. For all temperatures of sintering, the thermal shock, whether it is due to exposure to an elevated temperature or due to a sub-ambient temperature, may be risky for the long term use of the MMC. This may be due to the fact that such exposures when the MMC is meant for long term use may affect either the strength properties or the ductility properties or both ductility and strength properties.

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